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Topics of the Month

Effluent plants and the budget

AT this time of the year it is customary for the Federation of British Industries to make representations of Budget policy to the Chancellor of the Exchequer. One question raised this year is of especial interest to the chemical and process industries. It concerns effluent treatment plants. It is pointed out that the increasing necessity for industry to install such plant and equipment emphasises a defect in the basis of depreciation for tax purposes of expenditure incurred to this end. The present position is that a depreciation allowance is given on so much of the installation as ranks as plant, but by far the greatest element in the cost of such installations lies in the installation of pits settling tanks and underground piping. In relation to such expenditure the basis of depreciation is severely inadequate. An allowance is given on the cost of the brick or concrete lining of pits for example, on the basis that this is expenditure on a building, but no allowance whatsoever is given on the costs of excavation which is a heavy, and indeed the major, element. The Committee on the Taxation of Trading Profits (the Millard Tucker Committee) considered the question of expenditure on 'cutting and tunnelling' generally. In the light of the general considerations it came to the conclusion that, owing to the difficulty of knowing, in many cases, whether the 'asset' created would depreciate, the correct course was to give no initial or annual allowances but to give a balancing allowance when it is clear that the expenditure has become valueless (para. 269).

This recommendation has not yet been implemented. It is believed, however, that, in the special circumstances of effluent treatment plant, the reasons for deferring all allowance into a balancing allowance do not apply. The nature of such plant is such that frequent renewal of much of the underground piping, etc., is required, which in turn necessitates excavation, while the obsolescence factor is high, necessitating redesign.

The F.B.I. has urged that the depreciation position of effluent treatment plant should be examined with a view to agreeing a realistic allowance on the full cost of an installation.

Chemical engineering triumph

THE first reasonably complete account of the creation of Britain's atomic energy industry, an industry worth scores of millions of pounds and employing thousands of workers in three great factories, is told in 'Britain's Atomic Factories,' a 5s. book published a few weeks ago. Chemical engineering skill of a high order was needed throughout to ensure the success of these vast undertakings. Take, for instance, the story of Windscale works, which has given Cumberland a new industrial centre. Here plutonium is produced from uranium in two atomic piles and afterwards separated from the uranium in a gigantic chemical plant.

These piles, with their two 415-ft.-high chimney stacks, each weigh some 57,000 tons and, to prevent earth subsidence, are built on a concrete mat 10-ft. thick and measuring 200 ft. by 100 ft. The fundamental chemistry on which these great plants and the processes used in them are based was worked out by a team of chemists under Dr. R. Spence, now head of Harwell's chemistry division, who had only 20 mg. of plutonium available for their investigations! It was from the results of this work, based literally on a pinhead of plutonium, that the solvent extraction process now used for separation on a large scale was evolved.

Added to the inherent problems of building a complex chemical plant, the designers of the separation plant had to contend with radioactivity. Because of the intense radioactivity from the uranium removed from the pile, the separation plant had to be built so that, once production had started, it could go on working throughout its life by remote control. There could be no possibility of subsequent repairs, adjustments or even normal maintenance work. The plant, in fact, is totally enclosed in a thick concrete shell, pierced only by measuring instruments. It has no pumps, valves or other apparatus liable to break down. It consists simply of tanks, columns and miles of connecting pipes, all built of a new type of stainless steel specially developed. Five thousand workers and 300 professional staff were needed to build Windscale, which covers 300 acres.

Uranium processing

WINDSCALE is one of the three atomic factories controlled from the headquarters at Risley, Lancs. The first to be built was the uranium factory at Springfields, Lancs.

At the time when the site was first chosen—in 1946—only laboratory work on uranium refining had been carried out and there was no time to build a prototype pilot plant. Yet the Springfields factory came into production in October 1948, barely 2½ years after the first plans were made. The plant, where uranium metal is dissolved from the powdered ore and cast for use in atom piles, is probably the cleanest in Britain. Because of the exceptional standards of purity needed in atomic materials and because uranium dust could be dangerous to workers, all buildings and plants are thoroughly cleaned after every shift. Elaborate ventilating systems are installed and all staff and visitors must change into spotless overalls, caps, gloves and boots before entering process areas. Many thousands of uranium rods, canned in aluminium, have been produced at Springfields since production began.

At the third factory built by Risley, at Capenhurst, Cheshire, natural uranium, which consists of a mixture of two isotopes of the metal, is converted to a gas and passed through thousands of filters so fine that they can distinguish between atoms of almost identical weight. This is to separate the fissile type of uranium from the non-fissile type. The plant includes many miles of pipes, connected by many thousands of joints, all of which, with the compressors, filters and controlling instruments, must be sealed against the tiniest leak of air and evacuated as carefully as a wireless valve.

In addition the plant must be kept always at tropical temperatures to prevent the uranium hexafluoride gas from solidifying.

The section in the book on health precautions will allay any anxiety that might have been felt about dangers from radioactivity. Owing to the precautions taken, workers in Britain's atomic factories are safer from 'occupational hazards' than workers in any other industry.

With its primary task of constructing Britain's first atom processing plants completed, the Risley organisation is now turning its attention to the production of electric power from the atom. An experimental atomic power station is already being built as the forerunner of others which will one day play a vital part in meeting the needs of industry.

'Britain's Atomic Factories' ends with the words: 'As much adventure lies ahead for Risley as lies in the past.'

Potash from the sea

A METHOD of producing potash from sea water has been discovered by Norsk Hydro, the Norwegian chemical concern. To exploit the method Norsk Hydro, together with the Dutch chemical concern, Mekog, have built an experimental factory near Amsterdam with an annual capacity of about 1,500 tons of potassium salt. If production at this factory proves successful, factories for the production of potash from sea water on a major scale will be built in Norway and Holland. Norway at present imports about 45,000 tons of potassium p.a. at a cost of £1,600,000 and Holland imports 160,000 tons costing about £8 million.

Norsk Hydro and Mekog have formed a special company, Norduco, to run the experimental plant and to make patenting arrangements, but subsequent commercial production in Norway and Holland will be the separate responsibility of the two parent companies.

The method has been worked out by the head of Norsk Hydro's research laboratory, superintendent engineer Jakob Kielland. The potassium salt which primarily will be extracted from sea water by the Norduco process is potassium nitrate. As sea water contains only on the average 450 g. of potash per cu. m., tremendous quantities of water are required. To produce a ton, 2,000 to 3,000 cu. m. of water are needed.

Potash, of course, is one of the most important fertiliser materials with a world consumption of about 2 million tons 20 years ago, 3 million tons 10 years ago and almost 5 million tons a year now. Mostly it is mined from potash salt deposits, the Stassfurt deposits in Germany being the biggest. In America potash is obtained from the brines of Searles Lake, California, and in Israel from the Dead Sea. In Britain there are deposits in north-east Yorkshire, but it is not yet known if they can be mined economically.

Cement research creates a new industry

ONE of the difficulties facing the builders of the Jinja Dam at Owen Falls in Uganda was lack of a locally made cement. By the time cement has been shipped out to Mombasa and brought up country by rail the cost is more than doubled. The problem has now been solved by research at the Building Research Station, D.S.I.R. A cement works, based on processes discovered at the B.R.S., has been opened at Tororo, Uganda. Its annual output is 60,000 tons and it is considered to be one of the most important steps in the industrialisation of the country.

Local raw materials for cement production would normally have been rejected. The limestone available contains phosphates which, for reasons which were not understood, makes cement produced from it a weak and badly-setting material. The Building Research Station was consulted by the Uganda Development Corporation. After carrying out

Comical Engineering Terms

"TOWER PACKING"

some investigations, the station was able to show how the phosphates affected the cement properties and how to get over the difficulties. This made cement manufacture possible and the building of a cement works could go ahead.

Good-quality cement can be made from the local limestone with up to 2% phosphate content. A second process developed by the station makes it possible to remove the phosphate entirely from the limestone. As phosphate is a useful fertiliser, a valuable by-product could be obtained.

Her Majesty the Queen is to open the Owen Falls Dam on April 29.

Natural glycerin versus synthetic

THE soap-synthetic detergent battle in the U.S. is profoundly influencing the glycerin industry. Synthetic glycerin production is increasing as soap manufacture declines. However, natural glycerin production is showing surprising resilience. E. Scott Pattison, manager of the Glycerin Producers' Association, has sent us information which shows that in 1953 output of glycerin from fats increased by 8 to 10 million lb., compared with the year before. Total glycerin production last year (on a 100% basis) was 218 million lb., 30 million above 1952. It is true, however, that synthetic glycerin output increased more than that of the natural product.

The surprising increase in natural glycerin output in the face of falling soap production is due to two factors: (1) use of better-grade fats in soap making and a proportionate increase, relative to total pounds of soap produced, of higher grades where glycerin yields are highest; and (2) greater production of glycerin as a co-product of fatty acids and fatty alcohols. Nevertheless, the continuing decline in soap production must ultimately reduce natural glycerin output, and Mr. Pattison predicts that, if over the next three or four years soap making falls at the rate of the past few years, glycerin from this source will fall below the level of 100 million lb. annually. An increase in glycerin from industrial fat-splitting and fat-based detergents must also be considered, however, and Mr. Pattison believes that output will not fall much below 125 million lb. p.a. in the next few years. Total glycerin output, however, he considers will reach 250 to 275 million lb. some three or four years hence and this implies a rise in synthetic glycerin capacity to nearly 150 million lb. p.a.

New process conserves scarce coking coal

EXPERIMENTS have been undertaken at the Fuel Research Station, London, to find to what extent low-rank coals of Types 400 and 500 can be used in coking practice in blends, in replacement of a proportion of the good-quality coking coals of Type 301. This problem is of the highest national importance, since the very limited reserves of Type 301 coal in this country are being rapidly exhausted and their production costs are relatively high.

H. Bardgett, who described this research in a paper given before the Institute of Fuel last month, stated that the experimental work was done in two Woodall-Duckham intermittent vertical chambers of mean width 11 in., heated by producer gas from a built-in producer. Comparative tests carried out before the war on cokes from this plant had shown that they were similar in properties to cokes produced from the same coals carbonised in coke ovens, and consequently there should be no difficulty in applying the results of experiments in the intermittent vertical chambers to commercial coke-oven practice.

The experiments have shown that a weakly caking high-volatile Northumberland coal can be blended with Durham coking coal, gas-coking and gas-making coal, Types 301, 401 and 501, to the extent of up to 60% for producing coke, the shatter strength of which is appreciably better than that of a high proportion of coke produced and used in this country for metallurgical purposes. The replacement in this way, by Northumberland low-rank coal, of 40% of the 7½ million tons p.a. of Durham coal used for coking would represent a saving, allowing for additional coal to offset a 12% reduction of coke output, of up to about 2½ million tons p.a. of good coking coal.

Experiments have also shown that Durham gas coal, Type 501, can be blended in any proportion with Durham coals of Types 301 and 401 and produce coke of a suitable shatter index.

Reduction of coking rate within the limits 1 to 0.7 in./hr. has been shown to improve appreciably the shatter strength of coke produced from the particular coals and blends used.

Durham low-volatile 'cinder' coal, which has been affected by igneous intrusion, and appreciable reserves of which exist, can be utilised in the production of metallurgical coke. Blended with Durham Type 401 coal to the extent of 20 to 25% it has little affect on the coke strength and yield, and the proportion of large coke produced is appreciably increased.

Peroxides from gum turpentine

THE new process for producing pinane hydroperoxide from gum turpentine, which was briefly reported in our February issue (p. 69), has four main steps: Hydrogenation of the turpentine to give pinane; purification of the pinane, usually by simple distillation; oxidation of the pinane with molecular oxygen to a peroxide content of about 50%; and stripping the oxidate under vacuum to recover the unoxidised pinane, leaving the pinane hydroperoxide as a residue. Starting with 8-lb. batches of turpentine, this process yields 4-lb. batches of peroxides about 80 to 90% pure.

In the basic research through which the practical development was reached, chemists prepared a pure form of pinane hydroperoxide and established its chemical structure as the *cis*-1-pinane 2-hydroperoxide.

The development of new products from components of gum turpentine would provide a promising approach

towards extending markets for pine gum, which need stabilisation against rising competition from new chemical industries. Production of hydroperoxides from gum turpentine would represent a large outlet. Application of turpentine hydroperoxides as polymerisation catalysts in the manufacture of about 700,000 tons of 'cold' rubber, for example, would use about 300,000 gal. (5,000 barrels, or 2 million lb.) of gum turpentine.

Turpentine's composition offers a wide opportunity for exploration by chemists seeking to develop new industrial products from pine gum. Components are primarily terpene hydrocarbon compounds having 10 C-atoms and 16 H-atoms. In American gum turpentine about 95% of the components are *alpha*- and *beta*-pinane; remaining constituents are other terpenes and other classes of compounds.

More information is available from the Naval Stores Station, Olustee, Florida, whose scientists developed the process.

Acid process raises nickel and cobalt yields

THE vast nickel and cobalt ore reserves at Moa Bay, Cuba, are to be worked by a new process developed by the Chemical Construction Co. and licensed to the Freeport Sulphur Co. The process is only now entering the pilot-plant stage and technical details are not yet available. However, we are informed by Freeport that the process involves sulphuric acid leaching of the ore and permits not only a high recovery of metal values than earlier processes but enables the nickel and cobalt to be obtained as metals. It is planned, eventually, to produce 30 million lb. of nickel metal and 3 million lb. of cobalt metal annually. The Moa Bay reserves contain at least 40 million tons of nickel and cobalt ore.

Moa Bay is on the north-east coast of Cuba, several miles east of the Nicaro nickel project, which is now being operated for the U.S. Government by another company. The ore for this project is being supplied by a Freeport subsidiary, Nicaro Nickel Co., which designed, built and operated the plant for the Government in the last war. Recently the U.S. Government announced plans for spending $43 million on enlarging its nickel plant at Nicaro. We understand that it is also interested in the latest Freeport development.

Improved ammonia process

SEVERAL design innovations are being incorporated in two new ammonia plants currently under construction by the M. W. Kellogg Co. in the U.S. A feature of the process is a re-forming step which takes advantage of the high pressures at which natural gas is available. By conserving the potential energy of the feed and producing synthesis gas at relatively high pressure, savings in compressor horsepower of 25 to 35% are obtained. A new design of reactor also contributes to the improved ammonia yields and lower operating costs. The Kellogg ammonia process is briefly as follows:

The first step is a re-forming furnace which converts about 70% of a natural gas feed into raw synthesis gas. This mixture is then charged to a combustion chamber where nitrogen in the form of air is introduced into the stream.

The heat of combustion of the oxygen in the air supplies the energy required to re-form the remainder of the natural gas and to elevate the gas temperature to 1,700°F.

As the mixture leaves the combustion chamber it immediately enters a quenching vessel where its temperature is lowered by direct water quenching. This not only brings the temperature to the required shift reactor temperature but also provides the steam required in the shift reaction.

From this point the mixture is fed to a gas purification section, the first step in which is a shift reaction. This converts the CO in the stream to CO_2, simultaneously producing H_2 as a result of the reaction with water in the form of steam.

Carbon dioxide removal follows, employing a regenerative M.E.A. system. This method of removing CO_2 is claimed to have five advantages: no oxygen contamination, negligible synthesis gas loss, essentially complete removal of CO_2, the production of CO_2 in a pure usable form, and compactness of equipment.

Following this purification operation, traces of carbon monoxide left in the stream are removed before passing the synthesis gas through oil separators where it mixes with a recycle stream from the primary separator. Thence it passes through the secondary separator which knocks out ammonia contained in the recycle. The synthesis gas is then passed through exchange and charged to the catalytic converters.

The product gases from the converters are cooled in water coolers and then by exchange with converter feed gas in the recycle gas exchangers. Anhydrous liquid ammonia is separated out in the primary separator and, after further cooling to 25°F. in the refrigeration system, goes to an anhydrous ammonia product flash drum.

Sulphite cellulose discovery

THE Stora Kopparbergs Bergslag Co., one of Sweden's most important exporters of pulp and newsprint, has evolved a method of using Swedish northern pine, instead of the conventional spruce, for the manufacture of sulphite cellulose. The work has been done at the company's Skutskär mill.

Dr. Ragnar Söderquist, general manager at Skutskär, states that the experiments have been going on there ever since 1925 and, indeed, it is a problem which has been engaging the attention of forestry and industrial chemists all over the world for many years.

The importance of the discovery is of first magnitude to Sweden. Inasmuch as her spruce is getting more scarce and is more expensive than pine, it will make possible a considerable saving. The new product is stated to be of very good quality, white and strong, and to have good printing properties.

The Stora chemists have arrived at the new method by starting with the use of sodium bisulphite instead of the previously used calcium bisulphide, and the process implies complete degradation of the refractory pine resins.

Although some progress has been made by other companies in the production of sulphite cellulose from pine—*e.g.* a U.S. firm is reported to have started making sulphite cellulose from Georgia pine, which is a different type of wood—it is believed that the Swedish company is alone in having successfully produced this product from northern pine.

As a result of the new discovery and its adoption at the Skutskär mill, it will be possible to switch an additional quantity of spruce, corresponding to nearly 45,000 tons of newsprint annually, to the Stora's newsprint mill at Kvarnsveden, and a new paper-making machine, capable of utilising this quantity, will be installed at that mill, which will then have an annual total capacity of 165,000 tons.

EXTRACTIVE CRYSTALLISATION WITH UREA

A new way of purifying 'difficult' substances

By E. V. Truter, Ph.D., A.R.C.S.

The ability of urea to form crystalline complexes with many types of straight-chain aliphatic molecules offers a new and elegant method for the purification of suitable substances. In the following article the structure and properties of these urea complexes are described and the fundamental principles governing the separation of mixtures is discussed. Extractive crystallisation is attractive from the engineering point of view because only simple equipment is needed for large-scale operation. The author concludes with a brief review of the applications of the process, e.g. *processing of fatty acids, purification of wool grease and sperm oil, the upgrading of petrol, and the refining of jet engine fuel to lower the freezing point.*

THERE are two general methods for purifying individual substances and for separating the components of mixtures: distillation and crystallisation. For a specific problem these may be used in combination, or either by itself may suffice. Each method, however, has clearly definable limitations. Thus it is a prerequisite of crystallisation that the substance be a solid at the temperature of operation; likewise, for successful distillation it is essential for the material to be stable at its boiling point. It is true that there are specialised techniques for the purification of substances which lie beyond the scope of these two general methods. For example, substances which are liquids at room temperature may be crystallised at -70°C., while a similar extension of the range of fractional distillation to pressures as low as 0.1 mm. has been rendered possible by the development of the spinning-band column. Nevertheless, these techniques require special apparatus and skill for their operation and so belong essentially to the research laboratory.

Extractive crystallisation with urea (and thiourea) is useful for the purification of many 'difficult' compounds. For example, the physical state of the material, be it solid or liquid, is of little importance. Many separations that have previously been carried out by distillation or crystallisation

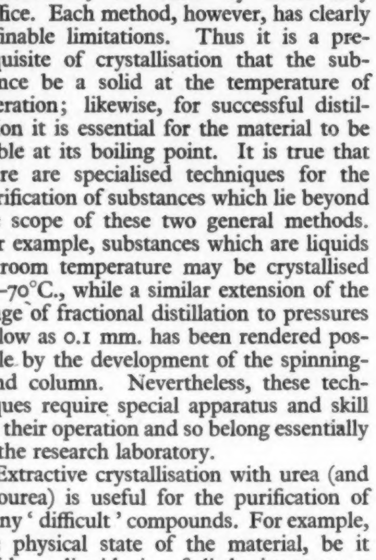

Fig. 1. The basic crystal lattices of (*a*) the urea complexes (hexagonal) and (*b*) the thiourea complexes (rhombohedral). (After Schlenk[2].)

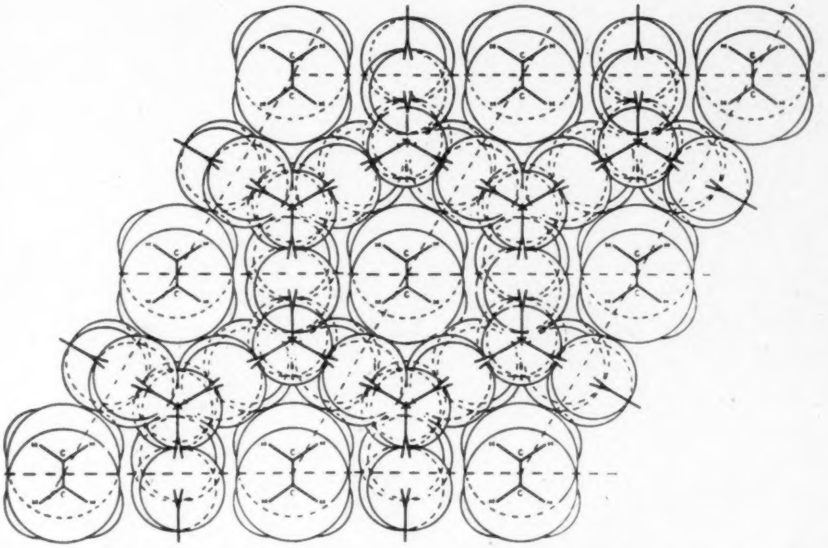

Fig. 2. Diagram showing the location of the endocytic molecule in the urea complex (after Smith[3]).

are more conveniently handled by means of the urea technique, while others have now become economically feasible for the first time. From the engineering point of view it has the great advantage that only the simplest equipment is required for large-scale operation.

The urea (and thiourea) compounds to be described here are entirely novel in their structures and properties, and must not be confused with the well-known complexes with metallic salts, *e.g.* silver chloride (thiourea is the active constituent of the recently marketed liquid dips for detarnishing silver), with acids, *e.g.* oxalic acid, or with the cresols (mixtures of *m*- and *p*-cresol are readily separated by means of their urea complexes).

In 1940, M. F. Bengen[1] was investigating the behaviour of milk proteins in the presence of urea. He found that any attempt to concentrate the solution by distillation resulted in the formation of troublesome foams, so he added *n*-octanol as an anti-foaming agent. After distillation, the residue was found to contain long, white, needle-shaped crystals which consisted of a complex of urea with *n*-octanol. Thus was discovered the first of what is now a very large series of urea complexes.

The structure of the urea addition compounds is best explained by reference to Figs. 1a and 2. In the urea molecule the unit $O = C\langle^{N}_{N}$ is flat, and these units are linked into ribbons by means of hydrogen bonds. The urea complex structure consists of a central organic molecule surrounded by three such ribbons arranged in a hexagonal helix (Fig. 1) and linked to one another

by a second series of hydrogen bonds. The result is an extended honeycomb structure in which the walls of the cells are formed by the urea molecules, and the contents of the cells are the organic molecules (Fig. 2). Although there are no discrete bonds between the endocytic (endon = within; cytos = cell) component and the urea molecules, the latter cannot form a honeycomb structure in which the cells remain empty. The presence of the endocytic component is essential, and if it is withdrawn the residual urea reverts to its normal tetragonal form.

In a structure such as this, where one component occupies channels of indefinite length formed in the other, the molecular ratio of the components depends only on the length of the molecules of the endocytic component. It is not surprising, therefore, that most complexes have non-stoichiometric molecular ratios. There are six molecules of urea in the unit cell, so that stoichiometric compounds arise only when the length of the endocytic molecule is a multiple of one-sixth of the unit cell length (Fig. 3).

The size of the triple helix formed by the urea molecules is fixed by the nature of the bonds which hold it together and, although a certain amount of distortion is possible, there is a definite maximum size which cannot be exceeded. Because there are no bonds between the organic component and urea, the factor which determines whether complex formation is possible is the cross-sectional diameter of the endocytic molecule. If the diameter is so large that the molecule cannot be accommodated in the lumen of the honeycomb, then clearly, complex formation is impossible. If, on the other hand, it can fit into the space available, then complex formation will result under suitable conditions.

Since the chemical nature of the organic component does not contribute to the process of complex formation, it is apparent that any straight-chain molecule will form a complex, provided that it adequately fills the channel. Thus, complexes of aliphatic hydrocarbons, ethers, alcohols, acids, esters, aldehydes, ketones, halides, nitriles, amines and with many substances containing two or even more functional groups are known. In each homologous series there is a minimum chain-length which must be exceeded before the organic molecule is able to form a urea complex. Above this limit all members form complexes. The minimum number of atoms in the chain which will permit complex formation to occur is not less than three and is usually more. For example, it is three for ketones (acetone), five for acids (*n*-butyric acid), six for hydrocarbons (*n*-hexane), seven for alcohols (*n*-hexanol) and eight for esters.

In Fig. 4 the cross-sections of various types of molecule are depicted. In each case the circumscribing circle represents the maximum diameter of the urea lumen, and the dotted circle represents its mini-

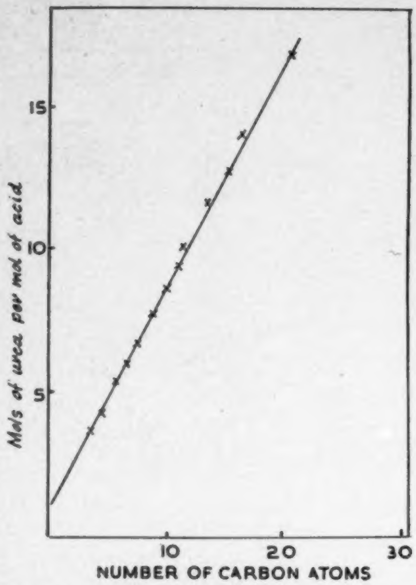

Fig. 3. The molecular ratio of urea to reactant is proportional to the chain-length of the organic molecule (the *n*-acids).

mum diameter. For complex formation to be possible the cross-section of the endocytic component must lie between these limits. It so happens that no long-chain compounds having diameters below the lower limit for urea have yet been tested, but it is to be expected that conjugated acetylenes, *e.g.* dimethyltriacetylene $CH_3—C{\equiv}C—C{\equiv}C—C{\equiv}C—CH_3$ will not form complexes. Hence, in the case of urea, only the upper limit is of importance. It is seen from Fig. 4 that substances having a branched-chain methyl group, or even a phenyl or cyclohexyl group, can be fitted into the lumen. Such substances can only form complexes provided that they contain a number of atoms larger than the minimum required for the series to which they belong. As the cross-sectional diameter at the widest part of the molecule approaches the maximum value permissible, so the ease with which complex formation occurs diminishes. Cyclic systems do not form complexes unless they are attached to long, unsubstituted aliphatic chains. For example, benzene just fits into the space available but does not form a complex spontaneously; *n*-octadecyl benzene, on the other hand, readily forms adducts. The critical limit for homologous series having side-groups is higher than for simple straight chains,[4] or looked at from another viewpoint, a substance with a side-group must have a long unbranched portion in the molecule to act as anchor.

Properties

The complexes of urea (and thiourea) are large white needles which may easily reach a length of 15 mm. They do not have definite melting points, although complexes of molecules containing less than about 16 carbon atoms undergo an apparent crystal transformation at temperatures which depend upon the rate of heating. The transformation temperatures must be measured using a hot stage microscope, and if the rate of heating is rapid the reproducibility is about ± 2°C. Complexes of longer organic molecules decompose at the melting point of urea.[5] Because the internal component can diffuse through the tubes in the structure, the stability of the complexes is determined by the vapour pressure of the endocyte; the higher the vapour pressure the more rapidly does the complex decompose. Thus, *n*-heptane/urea decomposes quite rapidly in the air, but oleic acid/urea is indefinitely stable.

The addition compounds are rapidly

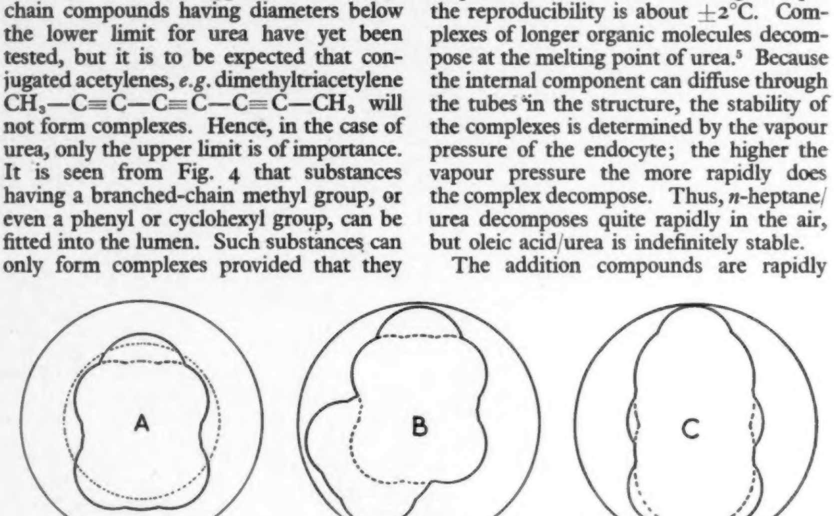

Fig. 4. Diagrams of the cross-sections of six branched-chain and cyclic molecules showing the extreme limits of the urea lumen: (*a*) simple ester; (*b*) methyl substituted ester; (*c*) phenyl ester; (*d*) benzoate; (*e*) benzene; (*f*) trimethyl substituted hydrocarbon.

decomposed on contact with water; the urea dissolves, leaving the organic component floating on the solution.

Thermodynamics of the urea complex

In the dissociation reaction for a complex of a single pure reactant: 1 *molecule complex* = 1 *molecule reactant* + *m molecules urea*, the dissociation constant may be written

$$K = \frac{a_r \cdot a_u^m}{a_c} \quad \ldots\ldots\ldots\ldots\ldots \text{(1)}$$

where a is the activity of the material indicated by the appropriate subscript. In order to measure K, which may also be regarded as a coefficient of partition of the reactant between the liquid phase and the complex, three methods may be used:

(1) **Aqueous reaction.** An aqueous solution of urea is mixed with the pure liquid reactant so that a second liquid phase is formed. When the system has come to equilibrium, the concentration of urea in the aqueous phase is determined. It is assumed that we are dealing with ideal solutions and that the concentrations are measured in mol fractions, so that in equation (1) as the reactant is present as a second liquid phase, $a_r = 1$, and as the complex exists as a separate solid phase, $a_c = 1$. Hence, from the relation between the activity of urea and its concentration (Fig. 5), the value of K can be determined.

(2) **Non-aqueous reaction.** A solution of the reactant in a solvent that cannot form a complex is poured on to and allowed to come to equilibrium with an excess of solid powdered urea. By judicious selection of the neutral solvent it can be arranged that the concentration of the reactant can be determined by measurement of the refractive index. If it is assumed that the activity is reasonably represented by the concentration, then in equation (1) $a_u = 1$ (excess of solid urea), $a_c = 1$ (solid complex) and therefore $K = a_r$.

(3) **Double concentration change.** A solution of the reactant in a neutral solvent is brought into equilibrium with an aqueous solution of urea, and the concentrations of the reactant and of the urea in their respective phases are measured. In this method, as in the aqueous method, it is necessary to know the composition of the complex.

These simple methods are equally suitable for use on the laboratory or industrial scale. If the second method is being used to prepare a complex it is advisable to moisten the powdered urea with water or methanol to catalyse the transformation of the urea structure.

Dewpoint measurements can also be used to determine the value of K, but the application is limited to complexes having a high vapour pressure of organic component. It is based on the fact that $K = p/p^0$, where p^0 is the vapour pressure of the pure reactant and p is its vapour pressure when in equilibrium with both complex and urea.

Once K has been determined the other

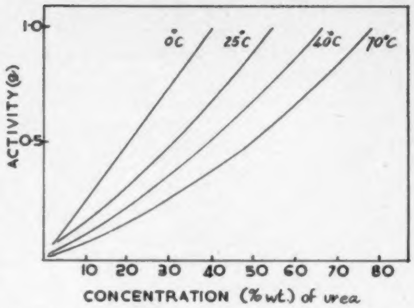

Fig. 5. The relation between the activity of urea and its concentration (data from Dunlop, et al.[6]).

thermodynamic functions may be calculated from the usual relationships:

$$-\triangle G = R.T.ln\ K = -\triangle H + T\triangle S$$

whence

$$ln K = \frac{\triangle S}{R} - \frac{\triangle H}{R} \cdot \frac{1}{T}$$

Complex formation is an exothermic process during which considerable rises in temperature may occur. This may be spectacularly demonstrated in the laboratory by adding a suitable reactant, such as n-decane, to a few grammes of powdered urea contained in a beaker and stirring the mixture with a thermometer.

In any homologous series the values of K and $\triangle H$ vary in a regular manner from one member to the next. The structural arrangement so dominates the properties of the complexes that it is possible, from only a few measurements, to set up linear equations from which the values of K and $\triangle H$ may be deduced for all members of the series. In the case of the normal hydrocarbons the equations are as follows:

$$\log K = 2.20 - 0.403m \text{ (at 25°C.)}$$
$$-\triangle H = 2.37m - 0.65 \quad \text{(Kcal./mol.)}$$

In order to evaluate these two functions the value of m (which is the number of mols of urea combined with one mol of reactant) may be calculated from the following equation:

$$m = 1.53 + 0.675n$$

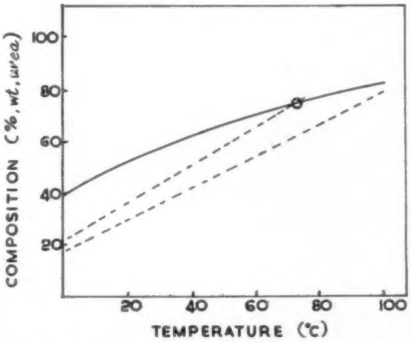

Fig. 6. Diagram showing the thermal stability of complexes of methyl oleate (*upper broken line*) and methyl stearate (*lower*) when in contact with aqueous urea solution. Full curve represents the solubility of urea.

where n is the number of carbon atoms in the hydrocarbon.

Less data are available for other compounds and only in the cases of the n-acids and the n-alcohols is it possible to set up similar equations. For the n-acids

$$\log K = 1.9 - 0.4m \text{ (at 25°C.)}$$
$$m = 1.08 + 0.71n$$

and for the n-alcohols

$$\log K = 2.5 - 0.43m \text{ (at 25°C.)}$$
$$m = 1.55 + 0.66n$$

For the methyl esters there are enough data to set up only one equation, *viz.*:

$$m = 2.2 + 0.66n$$

where n is the total number of carbon atoms in the ester.

A complex is stable in the presence of an aqueous solution of urea only when the concentration of the latter exceeds a particular value. The broken line in Fig. 6 shows how this varies with temperature, and the full line represents the solubility of urea. The two curves meet at a specific decomposition temperature above which the complex is not stable under any circumstances. If the decomposition temperature of a substance is below room temperature, then no complex will be obtained under normal conditions. This does not, however, exclude the possibility of complex formation at lower temperatures, or in the presence of suitable 'structure builders.' The observation that in each homologous series a certain minimum chain-length must be reached before complex formation can take place, is due to the arbitrary but very convenient choice of room temperature as the standard.

Mixtures

If, in a mixture, each reactant forms a complex which constitutes a separate solid phase, the system will behave like a series of independent pure reactants. In reality, however, the complexes form a single solid solution. If the activity of a reactant in the complex is equal to its mol fraction (y_i), ignoring the urea (*i.e.* the mol fraction is based only on the amount of endocytic component enclosed), and the mol fraction in the mother liquor is x_i, at equilibrium

$$K_i = \frac{x_i \cdot a_u^m}{y_i} \quad \ldots\ldots\ldots \text{(2)}$$

In the presence of excess urea $a_u = 1$, so that $K = x_i/y_i$ where $\Sigma y_i = 1$. Equation (2) shows that in a mixture the concentrations of the reactants in the mother liquor can fall below the values permitted for the pure reactants by themselves. Hence it follows that a substance which does not form a complex by itself may give a mixed complex in the presence of one that does, the latter being known as the 'structure builder.' Hence the separation of two substances both of which form complexes is not an entirely simple matter. The affinity of a substance for urea and

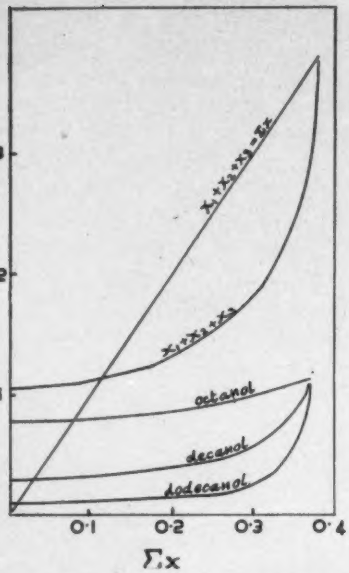

Fig. 7. Graphical solution of equation (7).

the stability of its complex (*i.e.* the value of K) is profoundly influenced by the chain-length and also by the cross-sectional area. The latter depends upon the number and nature of the substituents in the molecule, and also upon the number and distribution of unsaturated linkages. Although a clean separation of closely similar species may not be possible, considerable enrichment can usually be achieved. The degree of separation obtainable by a single urea treatment of a binary mixture can be deduced from equation (2) and is

$$\frac{y_1}{y_2} = \frac{x_1}{K_1} \cdot \frac{K_2}{x_2} \cdot a_u^{(m_1 - m_2)} \quad \ldots . \ (3)$$

If the values of K for each component are known, and also their initial concentrations, it is still not possible to calculate the concentrations of the reactants remaining in the mother liquor, nor in the complex, unless the activity of the urea at equilibrium is known.

It follows from equation (3) that when $m_1 = m_2$, that is when the molecules are of equal length, the factor involving the activity of the urea is eliminated. This is also true, irrespective of the values of m_1 and m_2, when there is an excess of solid urea present, *i.e.* $a_u = 1$. In these two cases the degree of separation is limited by the relative values of K. Generally, however, the molecular lengths of the two components will not be identical, so that if the activity of the urea falls below unity the degree of separation is altered in favour of the shorter molecular species. If the reactant with the greater molecular length belongs to a different homologous series and has a higher value of K, then use of a deficiency of urea improves the degree of separation. Consider, for example, the binary mixture oleic acid and palmitic acid: $m_o > m_p$ and $K_o > K_p$, so that a decrease in the activity of urea corresponds with an increase in the degree of separation.

When the two factors based on m and K are acting in opposition, it would appear that treatment of the mixture with an excess of urea is the best method for extracting the longer molecular chain. In practice, however, the practical requirements outweigh the advantages of maximum thermodynamic efficiency. Complexes of reactants containing more than about 15 carbon atoms are only slightly dissociated at room temperature, the mother liquor containing up to about 2% of the reactant. If a mixture of two such compounds is treated with an excess of powdered urea, only a small fraction of the total material remains in solution, and the ratio of the reactants in the complex differs only very slightly from that in the original mixture. A greater difference in the ratio of the reactants can be obtained only at the expense of decreasing the total amount of material extracted, *i.e.* by successive extractions with small amounts of urea.

In using the technique of extractive crystallisation, the problem of any separation may be stated in these terms. Given the mol fraction (z_i) and the K values for each endocyte in the initial mixture, and the composition of each complex, and also the mol fraction of neutral diluent ($1 - \Sigma z$), what are the mol fractions of the reactants in the mother liquor (x_i) and in the complex (y_i) after treatment with a given amount of urea?

It may be stated at the outset that if an excess of urea is not used the problem is insoluble unless the activity of the urea at equilibrium is actually measured.

The total amount of any reactant is given by the sum of the amounts in the mother liquor and in the complex, *i.e.* *mols present initially = mols in mother liquor + mols in complex.* To simplify the calculation all quantities are expressed relative to 1 mol of neutral solvent. In this way the mol fraction of neutral solvent initially ($1 - \Sigma z$), or in the mother liquor ($1 - \Sigma x$), represents one mol, so that the total number of mols present initially is $1/(1 - \Sigma z)$, and in the mother liquor is $1/(1 - \Sigma x)$. The distribution of any component throughout the system is then given by

$$\frac{z_i}{1 - \Sigma z} = \frac{x_i}{1 - \Sigma x} + y_i\left(\frac{\Sigma z}{(1 - \Sigma z)} - \frac{\Sigma x}{(1 - \Sigma x)}\right) \quad \ldots (4)$$

Now the equilibrium condition is stated in equation (2), so substituting for y_i in equation (4) we obtain

$$\frac{z_i}{1 - \Sigma z} = \frac{x_i}{1 - \Sigma x} + \frac{x_i \cdot a_u^{m_i}}{K_i}\left(\frac{\Sigma z - \Sigma x}{(1 - \Sigma z)(1 - \Sigma x)}\right) \quad \ldots . \ (5)$$

or

$$x_i = \frac{K_i z_i (1 - \Sigma x)}{K(1 - \Sigma z) + a_u^{m_i}(\Sigma z - \Sigma x)} \quad \ldots (6)$$

If excess urea has been used in the reaction then $a_u = 1$, but if less than the equivalent amount of urea has been used, *e.g.* a saturated aqueous solution of urea, then the concentration of urea in the solution must be determined before the problem can be solved. In equation (6) there now remain only two unknown quantities, namely x_i and Σx. But $x_1 + x_2 + x_3 + \ldots = \Sigma x$, so that

$$\Sigma x = \frac{K_1 z_1 (1 - \Sigma x)}{K_1(1 - \Sigma z) + a_u^{m_1}(\Sigma z - \Sigma x)} +$$

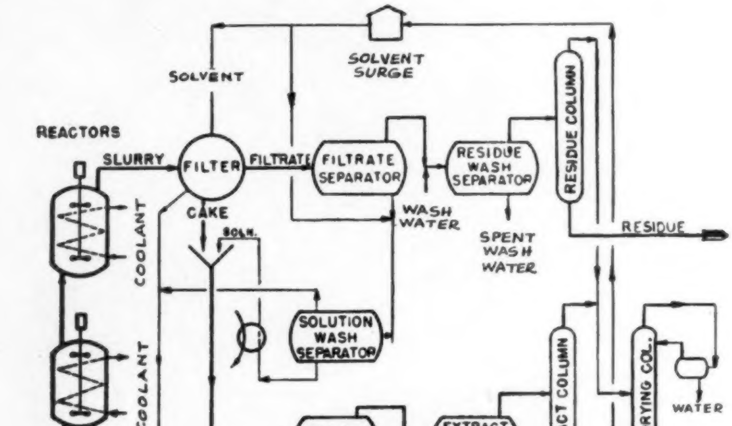

Fig. 8. Flow sheet of a continuous extractive crystallisation pilot plant with a throughput of 2 barrels/day of hydrocarbon. (After Bailey, et. al.[7]).

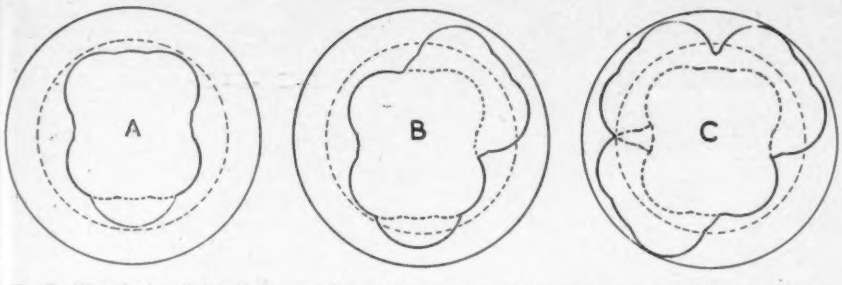

Fig. 9. The limits of the diameter of the thiourea lumen demand branched-chain molecules for successful complex formation: *(a)* **simple ester;** *(b)* **methyl substituted ester;** *(c)* **trimethyl substituted hydrocarbon.**

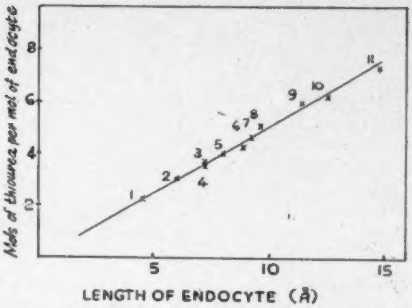

Fig. 10. The molecular ratio of thiourea to endocytic component is proportional to the length of the latter (data from Schlenk[2b]): *(1)* **cyclopentane;** *(2)* **1, 1, 1, 2-tetrachloroethane;** *(3)* **hexachlorethane;** *(4)* **tetrahydrodicyclopentadiene;** *(5)* **decalin;** *(6)* **p-menthane;** *(7)* **isobutylbenzene;** *(8)* **perhydroanthracene;** *(9)* **dicyclohexylamine;** *(10)* **2-bromo-octane;** *(11)* **p-dicyclohexylbenzene.**

$$\frac{K_2 z_2(1 - \Sigma x)}{K_2(1 - \Sigma z) + a_u^{m_2}(\Sigma z - \Sigma x)} + K_3, \text{ etc.} \quad \ldots\ldots\ldots\ldots\ldots (7)$$

The most convenient procedure for solving equation (7) is to use equation (6) as the basis for a separate computation for each component. Arbitrary values are assigned to Σx and the resultant values of x_1, x_2, x_3, etc., are calculated. Then, from a plot of Σx against $x_1 + x_2 + x_3 +$ etc., the unique solution is obtained. If the graphs of x_1, x_2, x_3, etc., against Σx are also plotted at the same time, the values of the former can be read off, and from them the corresponding y-values may be calculated.

Suppose, for the sake of illustration, that a ternary mixture of equimolecular proportions of *n*-octanol, *n*-decanol and *n*-dodecanol is to be subjected to urea extraction. The alcohols (0.2 g. mol of each) are dissolved in benzene (1 g. mol) and allowed to come to equilibrium at 25°C., with an aqueous slurry of urea (urea, 500 g.; water, 200 ml.). The K values at 25°C. are

n-octanol	0.341
n-decanol	0.089
n-dodecanol	0.022

also $z_1 = z_2 = z_3 = 0.125$

and $a_u^{m_1} = a_u^{m_2} = a_u^{m_3} = 1$.

The four graphs are plotted in Fig. 7 and the results are tabulated below:

	Initially		*Mother liquor*		*Complex*	
	mol.	%	*mol.*	%	*mol.*	%
n-octanol	0.2	33.3	0.0901	66.6	0.1099	23.7
n-decanol	0.2	33.3	0.0345	25.4	0.1655	35.3
n-dodecanol	0.2	33.3	0.0108	8.0	0.1892	41.0

Data for deciding the best procedure for carrying out a particular separation are still lacking. At the present time it seems as though there may be several equally good methods, of which the following example illustrates one possible mode of operation. Suppose it is desired to separate a mixture consisting of equal amounts of the methyl esters of stearic, oleic and linoleic acids (100 g. each). The esters are dissolved in hot benzene and poured on to urea (320 g.) which has been moistened with water (10 ml.) to catalyse the rearrangement of the urea structure. The mixture is stirred continuously while being maintained above 76°C. for about two hours. The solid complex is then filtered off at this temperature, it is decomposed by the addition of water, and the methyl esters are recovered. The benzene mother liquor is then poured on to more powdered urea (180 g.) moistened with water (20 ml.) and allowed to stand at room temperature for several hours. The complex is then filtered (or centrifuged) off, decomposed with water, and the esters recovered. The approximate compositions and yields of the three fractions obtained in these two operations are:

First urea complex: 95 g. composed of 99% methyl stearate and 1% unsaturated esters.

Second urea complex: 65 g. consisting of 6% methyl stearate, 86% methyl oleate and 8% methyl linoleate.

Benzene mother liquor: 140 g. which does not contain any methyl stearate, 31% methyl oleate and 69% methyl linoleate.

Thus, two treatments with urea make it possible to obtain methyl stearate of high purity (by commercial standards), together with an oleate-rich fraction and a linoleate-rich fraction. The latter can be concentrated even further by a third urea treatment.

The essential simplicity of the urea process makes it eminently suitable for either batch or continuous operation on a large scale. Bailey *et al.*[7] have set up a continuous extraction pilot plant with a throughput of 2 barrels/day of hydrocarbon. The flow sheet is shown in Fig. 8, and the operation is as follows. A saturated aqueous solution of urea is mixed with a solution of the hydrocarbons in methyl *iso*butyl ketone (MiBK); the ketone functions as a non-complexing, water-insoluble diluent for the hydrocarbons. The aqueous urea phase is kept near saturation by maintaining a thermal gradient through the plant. The warm solution of urea, together with the other reactants, is introduced into the reactor which is equipped with a stirrer and a cooling system. Urea complex is precipitated, usually in the form of fine acicular crystals, and is removed by centrifuge or rotary filter. The filtrate passes to a phase separator from which the MiBK solution, after it has been washed with water, is distilled to yield the solvent and the hydrocarbons which do not form complexes (labelled 'residue' in Fig. 8). At the same time the aqueous urea solution from the filtrate phase separator is washed with MiBK, and then it is mixed with the filter cake and the mixture is heated to decompose the complex. After the hot mixture has separated into two phases, the hydrocarbons which formed complexes (labelled 'extract' in Fig. 8) are run off, and the urea solution is returned to the beginning of the process.

In the continuous process it is necessary to ensure that the materials are given sufficient time to react. The bulk of the complex is fairly considerable and adequate accommodation must be provided; one barrel of straight-chain hydrocarbon yields complex occupying about 50 cu. ft. Hydrolysis of urea is not important because it is suppressed by its own hydrolysis product, ammonium carbonate, which does not interfere with the complexing reaction. Typical operating conditions are shown below:

Final reaction temperature	80°F.
Initial saturation temperature of urea solution ..	95°F.
Regeneration temperature ..	140°F.
Urea solution circulation rate	3 gal./gal. feed
Solvent circulation rate (MiBK)	0.7 " "
Reactor residence time ..	12 min.

Thiourea

The unexpected behaviour of urea in forming these non-bonded complexes prompts one to enquire whether thiourea may not behave in a similar manner. Indeed, it has been found that, not only are the same phenomena observed, but that the actions of urea and thiourea are, to some extent, complementary.[2, 8, 9] The structure of the thiourea complexes differs slightly from that of the urea complexes in that the arrangement of the thiourea molecules is rhombohedral (Fig. 1b) and,

more important still, the diameter of the lumen is greater. The lumen size is such that straight-chain aliphatic compounds are unable to form complexes because they are too small. It is essential for the organic component to have a branched-chain or a saturated-ring system; benzenoid compounds do not form complexes readily. The following short list of organic compounds illustrates the type of molecule that can form thiourea complexes: 2-bromo-octane, 2, 2, 4-trimethylpentane, *cyclo*pentanone, *cyclo*hexane, camphane, *iso*butylbenzene, perhydroanthracene, carbon tetrachloride and chloroform.

In the case of thiourea it is more difficult to determine by inspection whether a particular molecule is capable of complex formation. A potential endocytic molecule must have a cross-sectional diameter lying between 5.9 A and 7.6 A (Fig. 9) and the molecule should be fairly uniform in section, *i.e.* a compound which falls below the lower limit for most of its length will not form a stable adduct. A single methyl substituent in a straight chain is not usually sufficient to confer complexing properties on the molecule (Fig. 9b).

In every other respect there is a close similarity between the two pericytic agents. The complexes are prepared by the same methods, and they have the same general properties. Thus the molecular ratio of thiourea to reactant is a linear function of the length of the latter (Fig. 10); formation of mixed complexes and the phenomenon of structure building is observed. Anchoring of narrow groups and of wide groups by portions of the molecule which approach the ideal also occurs.

Applications

The most obvious application of extractive crystallisation is for the separation of a substance or a group of substances which form complexes from another individual or group that cannot. Separation of complex-forming substances into enriched fractions forms an even larger field of application and two examples have already been discussed in some detail. Less obvious is the use of a complex to protect a material that decomposes spontaneously in the air. For example, the auto-oxidation of oleic and linoleic acids is entirely absent when the acids are sealed in urea complexes.

Laboratory examples of potential applications are very numerous. Thus the free acids in acid-cracked wool grease (about 18%) are readily removed as urea complexes, leaving a neutral grease and at the same time providing a fatty acid stock suitable for soap preparation. Any attempt to neutralise the free acids and to extract the soaps with water results in the formation of emulsions that are so difficult to handle that the separation is not considered worth while.

In contrast to the behaviour of wool wax esters, those of sperm oil form urea complexes very easily, and they can readily be separated from the triglycerides which do not form complexes. The urea treatment is simpler and more efficient than the more usual method of pressing.

Improvement in the octane rating of gasoline can be obtained by treatment with either urea or thiourea. With urea the straight-chain molecules are preferentially removed, leaving the improved fuel uncombined, while with thiourea the more desirable branched-chain molecules are extracted[9] and the straight-chain material is left behind.

Urea treatment can also be used for the refining of jet aviation fuels.[10] In this case the most exacting specification is that the freezing point of the fuel should be below −76°F. The precise f.p. of a fuel, if it lies above about −80°F., is determined, almost exclusively, by the proportion of straight-chain molecules present. Simple distillation can give products which fulfil the specification, but only in yields of less than 50%. On the other hand, a single treatment with urea gives a suitable product in 85 to 95% yield (Table 1). Furthermore, by treatment with the predetermined amount of urea, the volume rejected can be cut to the minimum.

Table 1. The relative efficiencies of urea treatment and distillation for lowering the freezing points of jet aviation fuels

Volume rejected	*Freezing points*			
	Sample A		*Sample B*	
	Distillation	*Urea treatment*	*Distillation*	*Urea treatment*
0%	−41°F.	−41°F.	−47°F.	−47°F.
5%	−45°F.	−52°F.	−57°F.	−63°F.
10%	−47°F.	−67°F.	−64°F.	−86°F.
15%	−49°F.	−82°F.	−65°F.	
50%	−51°F.		−78°F.	

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Advantages of cold welding aluminium

Encouraging laboratory tests and successful practical applications of the cold welding process on aluminium indicate that it produces a strong and true weld and is quicker and more economical than hot welding. Under proper controlled conditions, the simple application of pressure through a suitably-shaped die is all that is needed to effect a true weld. Promising laboratory experiments have been conducted by Bohn Aluminium & Brass Corporation, Detroit, Michigan. Patents for the process are available to those interested in employing or experimenting with the process.

Welds of various shapes and sizes, in both extruded and sheet aluminium, made and studied in the laboratory, show the versatility of punch design that is possible, according to an article in *Modern Metals*. The success of the weld is closely dependent on the correct relationship between the stock thickness of the part and the dimensions of the welding die. Both the depth of penetration and the width of the weld must be proportional to the gauge of the metal.

Two operations are required to make a cold welded joint: preparing and cleaning the parts; and applying the necessary pressure to produce the weld. For the latter, any press suitable of producing and controlling the required pressures may be used.

The actual welding is effected when metal is flowing away from the punch, exposing surfaces, free of oxide or other material, which readily unite. Additional pressure, after the metal has stopped flowing, helps to ensure a good bond.

A test of four samples of cold-welded 2S-0, 2S-H14 and 2S-H34 aluminium alloys showed that tensions at break ranged from 150 to 155 for the 2S-0, from 180 to 185 lb. for the 2S-H14 and from 190 to 200 lb. for the 2S-H34. All welds were made at 60,000-lb. load, with the same pair of punches making simultaneous impressions in both top and bottom. They had slightly rounded edges and an area of 0.02 sq. in. It appeared that the strongest joint was produced in the strongest alloy, but not in proportion to the differences in parent metal strength. Because of the inherent reduction of the area immediately surrounding the actual weld, failure will take place at the perimeter—indicating that the tensile values obtained were a minimum and the actual weld values much higher.

Much attention was given to a study of punch design. In comparing punches with slightly chamfered corners to those with sharp corners at 60,000-lb. load, simultaneous top and bottom impressions and controlled penetration, the results seemed to leave little to choose between the two. There appeared to be no significant difference between welds produced with straight-sided and tapered punches. The latter were generally of slightly greater area and the amount of shearing of the stock was less. Punches making simultaneous impressions from top and bottom produced the stronger welds. Both sets—those making impressions from the top only and those making impressions simultaneously from top and bottom—produced the same reduction in thickness.

SIZE REDUCTION

Coal, clay, rocks and ores, chemicals, constructional materials for comminuting machinery, improvements in equipment

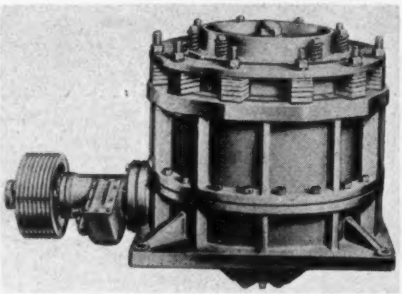
By R. V. Riley, Ph.D., B.Sc., F.I.M.

Theory and practice

RECENT literature on size reduction, although as voluminous as ever, has produced no important contribution to the fundamental theory of comminution. There was, however, considerable activity in the scientific study of practical crushing and grinding problems. A series of field tests on an industrial pulveriser were undertaken by the Department of Scientific and Industrial Research in collaboration with the Fuel Research Station.[1] The tests, so far as the results have been analysed at present, indicated that the present concept of grindability index for coals was not entirely satisfactory. The trials showed the necessity for a knowledge of the fundamentals of the structure of coal and the mechanism of its breakage. An alteration in the structure of coal by pre-treatment was found to materially alter its breakability.

K. Sedlatschek and L. Bass,[2] and also F. Weidenhammer,[3] have made contributions to the general theory of practical milling processes. A new textbook on chemical engineering[4] has a chapter on the elementary treatment of the unit operation of size reduction. E. L. Piret[5] has surveyed methods of recording screen analyses and energy requirements as derived from laboratory tests on the fundamental aspects of grinding. The results of crushing trials on many minerals and rocks were reported and critically discussed by T. Seguiti.[6]

A useful description of well-known methods of comminution indicating the equipment to be used for specified crushing and grinding operations has been given by H. W. Erickson.[7] Many types of grinding mills are in daily use in the chemical industry for coarse grinding, say 6 to 35 mesh, intermediate grinding, 35 to 70% passing 200 mesh, and for fine grinding, *i.e.* 70%, minus 200 mesh down to 10 microns, or even smaller. Erickson stated that these three categories of fineness should be balanced properly to secure the quickest yet economical operation. Open and closed circuit grinding, stage grinding, wet and dry grinding processes were all described and examples of these methods were given. The mills were classified in this article as revolving mills, rolls and roller mills, impact pulverisers, and attrition and Burnstone mills. Whilst this article was comprehensive, it lacked detail of the type contained in the more specialised work by C. E. Golson.[8]



[Photo: Sir W. G. Armstrong-Whitworth & Co. (Ironfounders) Ltd.

The 'Kue-Ken' gyratory crusher has especially low head room requirements so that it is adapted to underground working or for attaching to the discharge hopper from a wagon. The gyratory cone secures 'crushing without rubbing.' It is protected automatically against lubrication failure and against overloading. The working parts are sealed and work in oil. The degree of fineness is quickly adjustable by simply tightening several nuts at the top of the machine. The machine is made in two units, 18 in. and 36 in., which give a range of crushing performances.

O. H. Garlick and P. O. Abbe[9] have made a practical study of a laboratory jar mill in order to determine the operating conditions necessary to obtain efficiency. For dry grinding of ceramics in mills not greater than 36 in. diameter, it was considered that the best results were obtained with mill speeds from 55% to 60% of the critical centrifuging velocity. The solids in the mill should be between 25% and 33% of the total mill volume and the balls should occupy about 50%. In wet milling, there was an optimum consistency of slurry in which the balls or pebbles moved freely but without the tendency to either float or sink.

V. V. Tovarov[10] has studied ways for increasing the output of ball mills used for the grinding of cement. It was shown that careful classification of material after grinding to the desired size were important factors. The grinding of cement clinker was also critically examined by R. A. Loveland[11] who investigated ratios of ball load to clinker for optimum working. The laboratory grinding tests were discussed in relation to industrial closed-circuit grinding and to ball mill design. Similar studies on the ball milling of several ceramic materials were made by T. Tanaka and N. Saito.[12]

Information upon the vexed problem of deciding when to add new grinding balls to rotary mills has been given by K. Jacob.[13] It was shown that the grinding efficiency was lower when an arbitrary decision on 'topping up' was made and a mathematical calculation was put forward to indicate the exact time for ball additions. It often proves impossible to determine crushing efficiency exactly because of the difficulty of measuring the surface areas of the solids before and after crushing. S. Ergun[14] described a useful gas flow method for the determination of the geometric surface areas of crushed solids from measurements of pressure drop as a function of gas flow rate and bulk density.

Coal Crushing

Several papers on coal crushing have appeared. J. E. Littlechild[15] has discussed the need for coal blending in modern carbonising practice. The increasing scarcity of good coking coals makes it important to crush all coals for coking finely enough for them to be mixed and blended effectively. Dispersal of the mineral matter in the coal improves the strength of the coke. Thus grinding of coal is not only useful but necessary. In South Wales, coking coal is ground to 80% passing a $\frac{1}{2}$ mm. aperture. The crushing operations are divided into three stages:—

(*a*) Coarse crushing (unnecessary when coal has already been crushed at the washery).

(*b*) Intermediate crushing.

(*c*) Fine crushing (where necessary).

The author describes the advantages and disadvantages of the jaw crusher, toothed roll crusher, gyratory crusher and the Bradford breaker for coarse crushing. One important advantage of the Bradford breaker is that it is selective, enabling a reduction in the ash content to be made during crushing. For intermediate crushing, the hammer mill, cage disintegrator or roll crusher are suitable, and these give

a product which tends to lie between 10 and 30 mesh. For fine grinding when necessary, the ball, tube or rod mill, centrifugal grinder and attrition mill are suitable.

In America also,[16] great importance is attached to the blending of coals for coke production and every major undertaking regarded the fine crushing of coal as a necessity in order to obtain a constant bulk density. Coal crushing is usually carried out by the Bradford breaker and the Pennsylvania hammer mill.

S. Guruswamy[17] has tried to develop a method of assessing the random breakage of coal during mining and also in crushing operations. He recognised the need for a toughness factor which may be used to classify different coals. His toughness factor was defined as the average size, expressed as a percentage of initial size, of that part of the product containing all the fines and weighing 1% of the total.

Descriptive articles on coal crushing by claw hammer type machines are to be found in an official house organ of one well-known manufacturer of heavy crushing machinery.[18] Similar descriptive matter has been published[19] relating to roll crushers, swing claw crushers, jaw crushers and rotary drier equipment for coal, and also an article on the ball milling of coal.[20]

Clay preparation

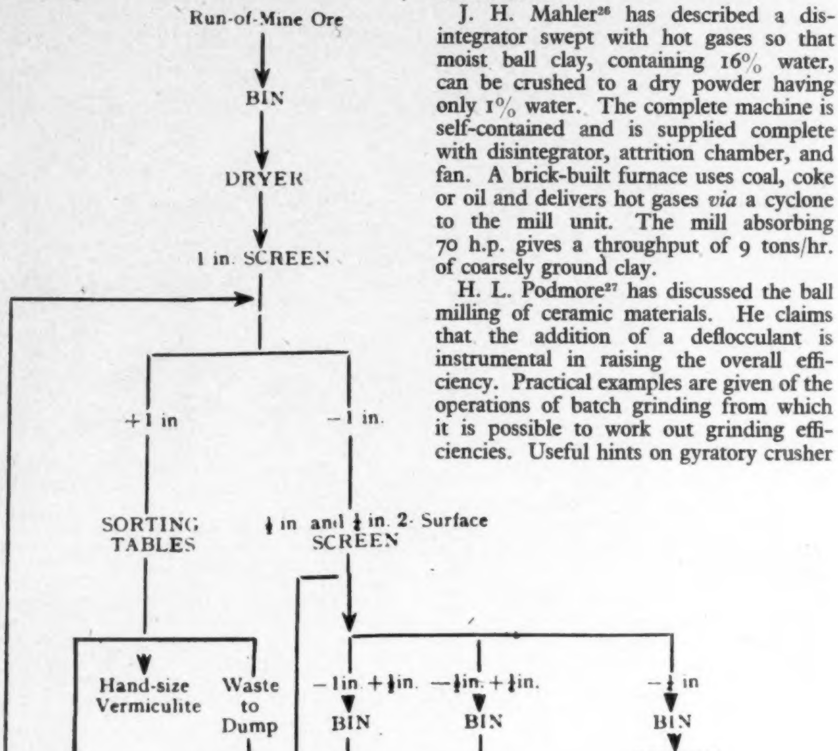
The ceramic and pottery industry depends for its continuance upon the economic utilisation of crushing and grinding machinery. A. Riley,[21] in an authoritative article, has described the methods used for the conditioning of flint pebbles and china stone. The latter is crushed in a two-stage process to pieces no bigger than ¾ in. diameter. The flint is first calcined and then put through a similar process of primary crushing. Finer grinding is accomplished by pan milling or by cylinder milling in the Hardinge mill. Finally, the crushed product is classified by a form of elutriation process. The breaking down of clay also calls for judgment in the correct choice of grinding equipment.[22] Much depends upon the hardness of the material, the size of the primary lumps, the amount of natural moisture, the impurities present, and the fineness of the end product. Very large lumps of clay direct from the mine are broken in roll crushers. Friable clays can be treated in the hammer mill. For rock-like clay bodies, jaw crushers, cone and gyratory crushers or dry pan crushers are more suitable. The jaw crusher can be used for large and hard lumps of clay provided the clay is dry.[23] For soft clays corrugated rolls are suitable. The pan mill must not be overlooked, but this is essentially a secondary grinder and cannot be expected to deal directly with the largest lumps from the mine.

Y. Shiraki and S. Kawanami[24] have described their experiences with the ball milling of Japanese china clays. The relations between grinding time, fineness and viscosity of the mill charge have been measured and the effect of the size of the grinding balls upon the grinding efficiency considered.

The action of an edge runner pan mill upon the grains of material in clay minerals has been explained by H. Reich.[25] The large grains or agglomerates are broken down under the miller, but the smaller particles are subjected only to gradual abrasion. In addition, there is a shearing action which results in the coating of the grains with thin films of water, thus improving the workability of the clay, a conclusion which justifies the reliance which has long been placed on the edge-runner mill.

J. H. Mahler[26] has described a disintegrator swept with hot gases so that moist ball clay, containing 16% water, can be crushed to a dry powder having only 1% water. The complete machine is self-contained and is supplied complete with disintegrator, attrition chamber, and fan. A brick-built furnace uses coal, coke or oil and delivers hot gases *via* a cyclone to the mill unit. The mill absorbing 70 h.p. gives a throughput of 9 tons/hr. of coarsely ground clay.

H. L. Podmore[27] has discussed the ball milling of ceramic materials. He claims that the addition of a deflocculant is instrumental in raising the overall efficiency. Practical examples are given of the operations of batch grinding from which it is possible to work out grinding efficiencies. Useful hints on gyratory crusher



Flow diagram of the crushing of vermiculite.

selection[28] are given. The power required for crushing was shown to be dependent upon many factors and a method was described of calculating the horse-power requirements from a knowledge of the hardness, toughness and specific gravity of the material, and the ratio of reduction and product size. Engineering details of milling maintenance have been given in a carefully written paper by H. M. Brown.[29] Similarly, notes upon magnetic equipment necessary to avoid damage to crushers working upon clay have been given by T. G. Hawker.[30]

Primary crushing of rocks and ores

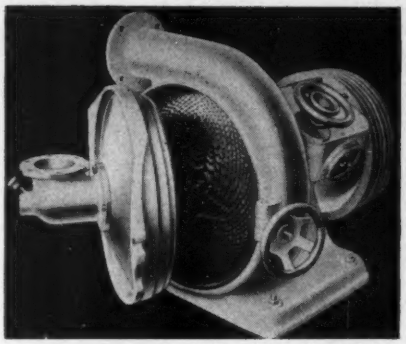
Many chemical products and most refined metals start life as ores, and it is in this field of ore winning and primary crushing that notable advances have been made. A new crushing unit has been installed at the new molybdenum mining project—Storke Level.[31] This is one of the largest gyratory crushers in the world. The crusher takes run of mine ore and crushes it to -9 in. The ore is then further reduced to -$\frac{3}{8}$ in. by cone crushers and this size serves as feed to the ball mills. The nickel mining industry in Canada has been driven to underground working in order to cope with the demand. Five huge crushers, each capable of crushing 450 tons/hr. ore, have been installed below ground at the International Nickel Co.'s mines in Northern Ontario.[32]

The primary crushing of iron ore by roll crusher in which lumps up to 4 ft. by 4 ft. are reduced to pieces 10 in. downwards is claimed to be an essential contribution in the drive to produce more British pig iron.[33]

A study of non-ferrous ore dressing was the purpose of a recent visit of European experts to the United States of America.[34] For intermediate coarse grinding, the rod mill is favoured but for fine grinding, the ball mill tends to be used in closed circuit with a classifier. The rod mill also appears to have replaced rolls for the handling of lead and zinc sulphide ore in a Canadian company's works.[35] The rod mill, whilst taking the same raw ore, crushes down to a size suitable for direct feeding to the primary ball mills. The normal rod load is 76 tons or 45% of the total mill volume. The mill is driven at 19 r.p.m. by 1,000 h.p. motor and gives an output of 400 tons/hr. The results of practical experience with the crushing of gold ores since 1941 are given in an article dealing with the Hollinger crushing plant.[36]

W. W. West[37] has described 'percussion crushers,' hammer mills in which both impact and attrition served to give the resultant size reduction. The aim was to obtain with alumina quartz and perlite a cubical product and a minimum of 'fines.'

In an H.M. Stationery Office report[38] a full account is given of the methods of winning and preparing vermiculite in South Africa. The flow sheet shows the combination of hammer milling, winnowing and roll crushing required to condition this unique mineral. In contrast with a material of the softness of vermiculite, a new diamond crusher is described by O. Fritsch.[39] It splinters the diamond by a milling action instead of by impact crushing. The whole system is maintained under a slight vacuum to avoid loss of diamond dust. The material from the crusher is then ground in an iron ball mill to improve the shape of the grains.



[*Photo: Lavino (London) Ltd.*

The Alpine Biplex grinder, a toothed disc mill, is manufactured for use both dry and wet in the rubber, leather, paper, cardboard, cellulose, rayon, wood fibre and cork industries. The toothed granulating discs are made from natural or synthetic stone of special construction in sizes 400 mm., 630 mm. and 800 mm. diameter. The grinder has several unusual features, such as: (1) Direct coupling without gears to a balanced shaft giving a vibration-free transmission of high power. (2) Hand wheel and locking device for accurate fineness regulation and a dial indicator for measuring clearance between discs to 1/10th mm. (3) Easy access to grinder by means of swing door. (4) Water-cooled discs. (5) Facilities for coupling two or more machines in series.

Size reduction of chemicals

Sugar cane crushing mills have been known for 500 years and were among the earliest machinery used in the food industry. Modern sugar crushing mills[40] consist of six strands of three rollers 37 in. diameter by 84 in. long. These achieve a juice extraction efficiency of 96% to 97%. The mill rollers are of cast iron grooved to increase grip on the sugar cane. Each of the six mills is powered by a 600 h.p. steam turbine which drives the rolls at 3 to 4 r.p.m. Whilst sugar has been extracted for very many years, the commercial extraction of proteins from crops not normally used for foods is new.[41] A mill suitable for recovering the succulent parts of leaves consists of a double beater, one arm working at a different speed of rotation to the other. A 20 h.p. motor driving this machine is claimed to produce up to 6 tons/hr. of useful ground material.

K. K. Majumdar[42] has described the preparation of Indian graphite. It is converted to a colloidal state by repeated grinding in a roller mill. Borax is disintegrated preferentially by means of rotary-toothed breaker.[43]

A new fine grinder known as the Intermediate *Atomill* is a useful extension of the range of grinders produced by a well-known British firm.[44] It will have a special appeal to the chemical, colour, cosmetics, food and plastic industries where batch operations are the rule and where frequent cleaning out between operations is necessary. The grinder may also be used continuously and will handle capacities 50% to 60% of those of the larger *Atomill*. It is driven by a 10 h.p. motor and the grinding action is obtained by ten hammers which pulverise the material against the heat-treated steel mill liners. A smaller grinding mill designed by another well-known company was produced for laboratory processing and control work.[45] Grinding is done by a carborundum stone revolving at high speed against a stationary carborundum stone. The power requirement is only $\frac{3}{4}$ h.p.

Another mill suitable for the comminuting of fine chemicals[46] had a swing hammer working against a series of interchangeable screens which grade the material down to 200 mesh. Provision is made for water cooling so that the mill is suitable for use with pharmaceuticals, including antibiotics.

For laboratory preparations, a three-tier jar mill has proved serviceable in the preparation of bacterial media.[47]

British Patent 647,282[48] covers a novel method of grinding wood.

B. Kopelman and V. B. Compton[49] have described a method of handling pyrophoric powders in organic liquids or in argon to avoid firing during grinding.

New constructional materials

H. Z. Schofield[50] has discussed the advantages of ball milling pure refractory oxides such as beryllia, zirconia, magnesia, etc., in mills in which the balls and liners are of the same material or similar to avoid contamination. Enamel frit makers have considered the merits and demerits of high density grinding media.[51, 52] A high density alumina ceramic material has been developed to meet these requirements. It has a specific gravity of 3.4 and is thus about 40% heavier than the conventional porcelain ball. The 'net effective working weight' of the balls submerged in enamel was, however, twice that of porcelain. The advantages claimed are that more balls may be included in the mill, different ball sizes may be used, and it is possible to increase the frit charge, so getting more output per hour.

Martensitic white cast iron balls appear to be becoming increasingly popular[53] in cement grinding. This may be due to the popularity of the alloy iron composition for liners[54] and also to the improved methods of die casting of balls to give a dense ball free from porosity.[55] Manganese steel for mill liners, whilst still frequently used, appears to be giving way to alternative irons and steels which can be worked more readily. 'Cromax steel'[56] can be cast to shape, heat-treated for

machining and finally heat-treated again to put it in a hard condition in which it will withstand the abrading action of the ball mill. Tungsten carbide[57] has found an application as a hard tip to the pulverising hammers in a swing hammer mill.

Improvements in equipment

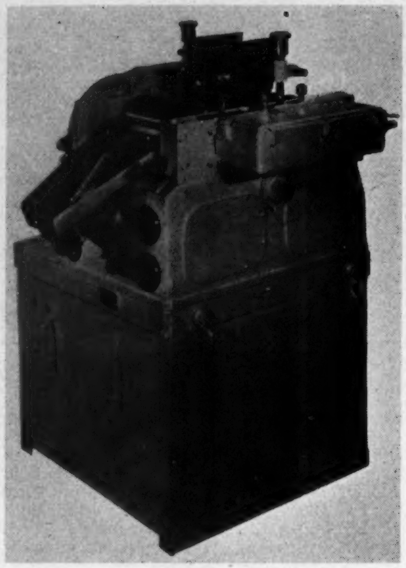
Primarily designed to produce sand to rigid specification, an end discharge rod mill was developed to give a minimum amount of fines.[58] A cubical-shaped particle was produced preferentially and under dry conditions the mill was well adapted to produce a −4 to −8 mesh product in one pass.

G. B. MacDeermid[59] has given the results of 3½ years' experience of rod mill operation in the crushing of ores. H. Mote[60] has claimed a method of discharging a ball or rod mill by pneumatic means in the continuous grinding of finely divided material.

A number of patents relate to mechanical improvements; A. F. Burgess[61] has claimed an improved design of gyratory crusher with a special bearing arrangement. B.P. 646,526 relates to a form of conical grinding mill with special discharge arrangements.[62] B.P. 646,686 describes a method of securing a resilient but firmly anchored upper ring which put the pressure on the balls in the crushing chamber of the ring and ball mill.[63] Improvements in general pulverising apparatus[64] and in a vibratory and rotary ball mill[65] have also been the subject of patents. B.P. 646,540 claims resiliently mounted delivery plate at the exit from a roller mill to divide the stream of crushed material.[66] A diamond pattern on the crushing face in order to reduce the bulging and distortion of jaw crusher face plates has been claimed by R. V. Baker.[67] A special thrust bearing to overcome the thrust between the toggle link and the eccentric that operates it has been claimed by H. H. Rumpel.[68] E. G. Loesche[69] has claimed an improvement in edge-runner mills.

Laboratory size jaw crushers and grinders have been described in trade literature,[70] as also have full size jaw granulators.[71] K. H. Suttor[72] has referred to two types of sieveless hammer mills. An interesting new development in rock crushing is the *Gyrasphere* crusher[73] which works upon the principle of an inverted pestle and mortar. There is a large feed opening and the design permits unobstructed and unlimited feed. A feature of this type of crusher is the greater freedom from liner wear even when crushing siliceous rock of great hardness and abradability.

In many chemical applications the term 'grinding' is often used to describe an operation which could more properly be considered 'dispersion.' The registered name *Dispersator*[74] is perhaps a happy combination of the words 'dispersion' and 'disintegrator' and refers to a machine of considerable value for the task of putting



[Photo: Pascall Engineering Co. Ltd.

The Pascall No. 3 model triple-roll mill has rolls 12 in. × 6 in. diameter and is driven by a 2½-h.p. motor. The latest feature is a new and simplified method of setting and adjusting the clearances between the rolls. Briefly, the principles of the new method are that the front and centre rolls are both movable in respect of the back roll, the centre roll sliding between the front and the back rolls. This model is suitable for the dispersion and milling of ointments, paints, enamels, creams, pastes and printing inks.

one liquid into an intimate suspension with another.

Other modern mixers and pot mills and also electro-vibro methods of feeding comminuting apparatus have been described.[75]

A new automatic control of feeding mechanism to work in step with the crusher is said to given an output of 20% to 30% more clay per day.[76]

Finally, mention should be made of the increasing popularity of the method of using the freezing power of solid CO_2[77] to facilitate the crushing of otherwise tacky or tough materials.

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EUROPE'S CHEMICAL INDUSTRIES

NEW GERMAN SURVEY

The rapid growth of the chemical and allied industries in Great Britain since the war has been matched by almost every other country in Europe. This fact is not always sufficiently appreciated and for this reason we present below a summary of chemical industry developments in 14 European countries. This information has been taken from a comprehensive review of Europe's chemical industries which appeared in the December 1953 issue of the German journal, 'Chemische Industrie.'

GREAT BRITAIN

THE development of the chemical industry in the U.K. has been greatly influenced by Britain's need to raise her exports to a higher level than ever before. The chemical industry's triple task has been to satisfy the demands of other export industries as well as the internal demand, and at the same time to increase its own exports considerably.

Development plans for the chemical industry envisaged investments of some £200 million for the period 1948-53. An interim balance drawn up in 1952 showed that, as far as investment values were concerned, 41% of this programme had been achieved and 34% of the projected facilities were under construction. About 15% of the expansion envisaged in the programme has been postponed, and 9% has been abandoned for various reasons.*

Exports of chemicals were increased from a value of £86.1 million in 1949 to 138 million in 1952. From January to September 1953 chemical products to a value of £94.9 million were exported.† The greater part of these exports have been sold in the sterling area, but the dollar markets have not been neglected.

One of the most remarkable developments in the British chemical industry is the manufacture of petroleum-based chemicals, which was begun in 1949. There are now four large plants engaged in this field, total investments amounting to about £40 million.

WESTERN GERMANY

Chemical production in the broadest sense has increased considerably in the past few years, but in 1953 there was a marked decline in prices. In the third quarter of 1953 the production index of the chemical industry was 156, compared with 131 for industry as a whole. Comparable figures for 1950 were 107 and 92 respectively. The price index tells a different story. In the third quarter of 1953 chemical prices stood at 184 against 218 for the rest of industry. The gap between chemical prices and prices generally has, indeed, widened since 1950, when the figures were 175 and 186, respectively.

The value of Western Germany's chemical production in the first half of 1953 totalled 5,264.9 million marks. Chemicals and chemical products to a value of over 1,000 million marks were exported. Imports were valued at 222.5 million marks.

The increases in output have been contributed by almost all sections of the industry. Here are examples, the output (in tonnes) of the third quarter of 1953 being compared with that of the same quarter in 1952 (in brackets): sulphuric acid (SO_3), 378,874 (329,070); hydrochloric acid (HCl), 27,807 (25,250); caustic soda ($NaOH$), 117,818 (90,327); methanol, 30,319 (15,741); cellulose plastics, 9,739 (7,126); pharmaceuticals (value), 241,512,000 marks (213,906,000).

FRANCE

According to the official production index, the production of chemicals in France is at present barely 50% of the pre-war level. But the index gives only an incomplete picture of the progress actually achieved. For example, it does not allow for new production started after 1938, nor for improvements in quality.

The production of ammonia, nitric acid and nitrogen fertilisers has doubled since 1938. The increase in sulphuric acid production, with a present figure of 1.96 million tons as compared with 1.4 million tons pre-war, is not so great. But here, too, great changes have been made in capacities and in the application of modern processes.

In electrochemical processing, the chlorine industry in particular has achieved notable advances. Production of calcium carbide has also greatly increased. The organic chemical industry has achieved particularly large increases in the production of methanol, formaldehyde, synthetic phenol, acetone and phthalic anhydride. In most of the new factories, production of organic chemicals is based on petroleum and natural gas, so that important basic chemicals such as ethylene oxide, glycol, ethanolamine, isopropanol, acetone, phenol and butyl derivatives have become available in large quantities at relatively low prices.

The petrochemical industry will have a favourable influence on the total production of synthetic detergents and, to a lesser extent, synthetic pharmaceuticals. A production of 100,000 tons of synthetic detergents is expected in 1954.

In plastics, the most notable recent achievements have been in the production of polythene, polystyrene and the silicones.

BELGIUM

Heavy chemicals comprise the main part of Belgium's chemical industry. The *per capita* production of sulphuric acid and nitrogen fertilisers is higher than in any other country. Belgian factories produce 5% of the world's nitrogen fertilisers.

During the last few years, steady expansion has taken place in the organic sector. Important products include plastics, organic pigments, coal-tar dyes and their intermediates, also pesticides, flotation agents for ores, textile intermediates and pharmaceuticals. In the dyestuffs field, specialities still not produced outside Belgium are chemicals for dyeing viscose stock. New products of the plastics industry include acrylic resins, silicones and plasticisers for vinyl resins. Since 1945, Belgium's pharmaceutical industry has developed several new products, including antihistamines, anti-rheumatics, X-ray contrast agents and analgesics.

Belgium is vitally interested in the abolition of import restrictions, since she must export over half of her chemical production.

DENMARK

The Danish chemical industry's extreme dependence on imports of raw materials led to a severe decline in production during World War 2. It was not until 1948 that production again exceeded the pre-war level, whereas Denmark's industry as a whole had already overtaken the pre-war level in 1946. Despite this, the value of chemicals produced in 1951 amounted to 1,400 million Kr.—12% of total industrial production. In terms of production value, the chemical industry is the third largest industry in Denmark. At 28,200 Kr. per worker, the net production value of the chemical industry is 1½ times that of industry as a whole.

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*These figures are now out of date. At the end of 1952, 75% of the 1948 expansion programme had been completed. In addition a new development programme has been embarked upon; this will cost £230 million in the period 1953-57. For more details see CHEMICAL & PROCESS ENGINEERING, November 1953, p. 368.

†Total value of exports for 1953 was £129,992,537, a decline compared with both 1952 and 1951.

The figures so far available for 1953 show an increase of 4% over the 1952 quantity of chemicals produced. In the first nine months of 1953 the export of chemical products decreased or stagnated, with the exception of pharmaceuticals, the export of which has risen by 50%.

The immediate future is not promising. With a contraction of markets, the Danish chemical industry would be confronted with strong competition, which it would have to fight on very uneven terms.

FINLAND

Finland's chemical industry is largely dependent on imports of raw materials and basic chemicals. The heavy chemical industry is founded primarily on indigenous ores, of which copper and non-ferrous ores are the most important. They constitute the basic raw materials for the production of sulphuric acid and superphosphates. Salt electrolysis has been steadily expanded during recent years, and supplies the chlorine necessary for Finland's important cellulose industry.

The pyrites-acid plants are no longer adequate to meet demand and a further increase in sulphur imports from the level of 30,000 tons p.a. will be necessary. In 1952, 20 plants were producing sulphite cellulose pulp, their annual capacity being 712,000 tons. In 1952, too, nine plants produced 443,000 tons of sulphite cellulose.

The paint industry has a capacity of 20,000 tons p.a. and fully meets domestic needs.

NORWAY

Cellulose and chemicals are the most important of all Norway's industries. In chemicals, the main emphasis is on nitrogen, production of which is for the most part in the hands of Norsk Hydro, the largest exporter of nitrogen in the world.

Co-operation between the cellulose and chemical industries is becoming closer from year to year. This is essential, if only to make the hitherto one-sided chemical industry less susceptible to crises. The sulphite spirit occurring in the cellulose industry is already being used on a large scale as a raw material for organic synthesis. The largest cellulose firm in the country, Borregaard A/S, obtains large quantities of acetaldehyde, acetic acid, butanol, ethylacetate and butyl acetate from its own sulphite spirit. Preparations are also being made for the production from acetaldehyde of octanol for the manufacture of plasticisers.

Considerable expansion has taken place in the young plastics industry. About 3,000 tons of urea resin for lacquers, varnishes and *Aminoplast* moulding compounds are produced annually. The urea required is supplied by Norsk Hydro, which produces 10,000 tons p.a. Formaldehyde is also produced indigenously, although the methanol required has to be imported. New plants for the production of PVC are being built and an output of 2,700 tons, part of which will be exported, is expected in 1954.

SWEDEN

The rich forests of Sweden supply the raw materials for her pulp industry, the bulk of whose products are exported. The hope of building up a chemical industry on large quantities of lignin obtained from pulping processes has not yet been realised, but the sulphite waste liquor is used to a large extent for the manufacture of alcohol, some of which is used for various organic products.

The annual consumption of sulphur, derived from pyrites and brimstone, is some 300,000 tons. The two largest consumers are the sulphuric acid and sulphite pulp industries. The greater part of the sulphuric acid production (about 350,000 tons p.a.) is used for superphosphate (about 500,000 tons p.a.).

Production of organic chemicals is based on calcium carbide and chlorine, which are manufactured electrochemically, as well as on wood alcohol.

HOLLAND

Although the chemical industry in Holland did not really begin until the turn of the century, today there are 1,600 plants employing 50,000 people. One half of their production is exported, representing 9% of Holland's total exports and 7% of the turnover of all Holland's industry. Since 1938, even ignoring the price increases that have taken place, the total turnover of the chemical industry has increased sixfold.

Owing to the importance of agriculture in Holland, there is a large superphosphate and nitrogen industry. The capacity for production of nitrogen fertilisers is today $2\frac{1}{2}$ times that of 1939. Sulphuric acid production, calculated as 100% acid, is 560,000 tons p.a.

Alkali electrolysis capacity has been increased to four times the pre-war figure, since the organic industries which use chlorine have expanded considerably. The production and processing of plastics is developing rapidly. Increase in production of chemical fibres, particularly rayon yarn, is striking. Output in this field is today $3\frac{1}{2}$ times as great as in 1938, and Holland is the world's second largest exporter, accounting for 16% of world exports.

ITALY

Italy has enough raw materials for the development of an efficient, highly productive chemical industry. True, the country is poor in coal, but this lack, which was felt so severely in the past, is being increasingly compensated for by the rich natural-gas deposits. Italy is also rich in sulphur, pyrites, bauxite, salt, mercury, arsenic, zinc and lead.

Despite the extensive damage to chemical plants during the war, it has been possible to exceed pre-war production in most of the basic chemicals. Production of nitrogen has been almost doubled. The proportion of ammonium sulphate in overall nitrogen production has been increased to about 60%. There were corresponding falls in the proportions of calcium nitrate, sodium nitrate, calcium cyanamide and ammonium nitrate. The increase in the production of phosphate fertilisers, if not so great, merits attention. The proportion of mineral superphosphates has decreased from 95% to somewhat over 90%.

Production of organic chemicals has reached a high stage of development in recent years, mainly because of the expansion of the petrochemical industry.

AUSTRIA

The development of Austria's chemical industry has been hampered by political events since 1918, and also by the absence of an industry supplying basic organic chemicals. The abundance of water power and highly developed sources of electricity should give Austria particular advantages in electrochemicals, but the exceptionally high cost of power restricts development in this direction.

Although the chemical industry's contribution to foreign trade lies chiefly in indirect exports, direct exports also show an upward trend.

The most important products are tar derivatives, plastics and synthetic fibres, and paints, varnishes and pigments.

SWITZERLAND

Switzerland is poor in raw materials and sources of energy, her only assets being water, salt, timber and cheap hydroelectric power. Raw materials and intermediates must therefore be imported. These circumstances oblige the Swiss chemical industry to concentrate on quality rather than quantity products—a policy requiring constant and intensive research.

The limited market at home compels manufacturers to export. Although there are 200 chemical firms, the industry is mainly in the hands of about a dozen large and medium-sized firms. Almost all of these have plants and sister companies abroad, through which they conduct a successful international trade in chemical goods, mainly dyestuffs and pharmaceuticals. In the last three years, exports of chemical products have averaged 15.1% of total exports.

SPAIN

The Spanish chemical industry has been built up in the post-war years without foreign financial help. The success achieved is reflected in the figures for 1952, when 7,500 chemical and allied firms with 11,500 plants produced 6,600 million pesetas' worth of chemical products. Inorganic heavy chemicals, particularly those from indigenous raw materials, form the foundations of the industry. Chief of these is sulphuric acid, of which some 800,000 tons p.a. is produced from pyrites. In 1952, Spain's production of sulphuric acid reached nearly half that of the U.K.

Equal attention is being given to the production of such organic chemicals as

are likely to improve the standard of living of the Spanish people, *e.g.* chemical fibres, plastics, pharmaceuticals, dyestuffs and surface coatings. Home requirements of surface coatings can already be met, while the annual production of organic dyestuffs amounts to 2,500 tons.

There is a strong tendency for chemical producers to amalgamate, and future development of the industry is likely to be largely in the hands of four concerns or groups.

PORTUGAL

Portugal's relatively new chemical industry is strongly supported by the State. The Government makes capital available for investment and ensures growth by granting tax exemption for 10 years. All new projects have to be approved by the Minister of Economics, who thus directs chemical production according to the needs of industry as a whole.

The country's sulphur requirements are met by the Alemtejo deposits and the production of sulphur compounds is an important industry. The fertiliser industry is able to meet the home demand, except for ammonium sulphate.

Facilities for producing organic chemicals and pharmaceuticals are being extended. The production of pesticides has recently been taken up by new companies.

The production of chemicals in Portugal is carried out mainly by a few large chemical firms, which are well equipped technically. There are numerous projects in hand for the further expansion of the chemical industry, based principally on exploiting agricultural waste products and indigenous raw materials.

Sicilian Superphosphate Project

By Our Rome Correspondent

A NEW factory to make superphosphate fertilisers is being built at Chaos Beach, near Porto Empedocle, Agrigento, S. Sicily. It will be operated by a new company of the Montecatini group which takes its name from the Greek colony, Akragas, that was founded in the vicinity more than 2,500 years ago.

The output of the new factory will contribute substantially to the development of Sicilian agriculture. The demand for fertilisers will increase with the completion of Sicilian land improvement schemes; for instance, an irrigation project near Agrigento will make a large area available for farming. Besides supplying home needs, Akragas also hope to export fertilisers to Egypt and the Middle East.

The new factory will produce superphosphate with 22 to 24% or 45 to 47% of P_2O_5, instead of the normal 18 to 20%. Superphosphate of these higher concentrations is becoming increasingly popular in many countries, particularly because of the resulting savings in handling and transport costs. This advantage will be particularly felt in Sicily, a hilly region where transport is a problem, and it will also improve the export possibilities of the product.

The factory site covers 86,000 sq. m. on the coast, adjacent to the railway line and close to the port. Before construction began, the site had to be cleared of unexploded mines and other relics of World War 2.

The factory is favourably situated, not only for the direct shipment of its products by rail and sea, but also for the importation of phosphates from Tunisia and Morocco. Another raw material, sulphur, is found near at hand in the Sicilian sulphur mines.

The capacity of the factory will be 50,000 metric tons p.a. of product with 22 to 24% of soluble P_2O_5, or 30,000 tons of one with 45 to 47%. Future extensions to give increased output of superphosphate are envisaged. Production of nitrogenous fertilisers is another possibility.

Port facilities are being increased and improved in anticipation of the increase in traffic which the factory will bring; the quay will be enlarged and loading facilities installed. The port traffic, which at present amounts to 100,000 metric tons of rock salt, 110,000 tons of coal, 30,000 tons of phosphates to supply a factory at Castelfranco, Italy, and 20,000 tons of gypsum p.a., will be increased by about 45,000 tons.

Cooling water for the factory is to be pumped from the sea at the rate of 5,000 cu.m./day. In addition the establishment will need 300 to 350 cu.m./day of fresh water, to be taken from the Voltano aqueduct. The enlargement of the aqueduct, at a cost of 1,200 million lire, is in hand. The provision of larger supplies of fresh water is being considered.

The administrative buildings will cover 1,970 sq. m., while the industrial buildings will cover 1,390 sq. m. More than 23,000 metric tons of cast iron, stainless steel, lead, copper, tin, antimony and vanadium, besides about 13,000 tons of cement, bricks, sandstone, refractory and other materials, will be needed for the project. The necessary plant and equipment has been ordered from Italian manufacturers, but some measuring instruments and the compressors for the oleum plant gases will be bought abroad.

The factory will have the following facilities (all tonnages in metric tons):

(*a*) **An oleum plant** with a daily capacity of 100 tons of concentrated acid. The equipment has been designed to Montecatini specifications.

(*b*) **Phosphate storage,** comprising five reinforced concrete silos each 10 m. in diameter and 21 m. high and each of 2,000 metric tons capacity.

(*c*) **A phosphate grinding plant** with two self-ventilated mills able to grind 16 to 17 tons/hr. of phosphate, corresponding to the quantity needed for the daily production of 30 tons of orthophosphoric acid and 170 tons of superphosphate with 22 to 24% of soluble P_2O_5.

(*d*) **An orthophosphoric acid plant,** designed for a daily output of 30 tons of P_2O_5.

(*e*) **A superphosphate plant** with a capacity of 10 to 12 tons/hr. and an installation for granulating the product.

(*f*) **Superphosphate storage and handling**—a rectangular area of 254 × 32 m. with weighing, bagging and loading facilities. The superphosphate will be conveyed by mechanical shovels to the grinding and feeding hopper of the weighing and bagging station. The filled bags will be mechanically sewn and prepared for loading.

The factory will be linked by 1,900 m. of rails with the main railway line. The connection with the quay will require 420 m. of rails.

The plant includes a sewage system discharging to the sea. Electricity will be supplied from outside and a transformer station is planned.

A building near the entrance, covering 650 sq. m., will include mess rooms and other facilities for the staff. On the other side of the entrance a building covering 240 sq. m. will house administrative offices, offices for technical personnel and a small chemical laboratory.

A mechanical workshop and a storehouse for provisions will be situated in a C-shaped building covering 900 sq. m. The nitrate and bags depot will be able to store 30,000 metric quintals of nitrate and 10,000 bags.

The factory is expected to be completed during the next two years.

Electrostatic precipitation. A 64-page brochure from Sturtevant Engineering Co. Ltd. is the tenth edition of their publication on electrostatic precipitation. In an introductory chapter readers are reminded that the precipitator utilises the general principles, known for many years, that if a gas conveying solid or liquid particles is caused to flow between two electrodes, one of which is charged to a high potential and the other earthed, then the gas and its burden are ionised and the particles or disperoids are driven to the earthed electrode, thus removing them from the gas stream. In the chemical and metallurgical industries the duties performed by these precipitators come under three headings: (*a*) collection of valuable dust or liquids, (*b*) cleaning of gases to prevent a harmful material being carried from one part of the plant to another and (*c*) the elimination of noxious odours. The brochure includes many photographs of installations in various chemical and cement works, for which a special design of precipitator has been developed. Diagrams demonstrate the working principles and features of design.

Health Hazards in the Plastics Industry

PLASTICS contain, in addition to the polymer, varying amounts of other chemicals such as catalysts, accelerators, stabilisers, hardeners, pigments, etc. Most polymers are chemically inert and under ordinary circumstances the final plastic material should be comparatively harmless. However, it is difficult in practice to effect complete polymerisation and traces of free monomer or other chemicals may be present which can sometimes produce diseases such as dermatitis.

Health problems in the manufacture and use of plastics are described by D. Kenwin Harris, of I.C.I. Plastics Division, in the *British Journal of Industrial Medicine* (1953, **10**, (4), 255-268).

Formaldehyde products

Formaldehyde and hexamine are well known as skin irritants, and many factories handling resin glues have had cases of dermatitis. A possible cause of this is that formaldehyde glues and resins will adhere to the skin and rapidly harden on contact with it. However, the main products considered in this paper are moulding powders or aqueous glues and resins which are only partially polymerised by intention so that they do not harden on the skin and are easily removed by washing. This may account for the comparatively low percentage of cases of dermatitis in spite of the fact that the products may contain substantial quantities of free formaldehyde. The addition of a hardener to these aqueous glues and resins is generally necessary before they are used commercially as adhesives.

In a group of factories making these partially polymerised glues and moulding powders, three types of dermatitis have been distinguished. In the first the patient has usually worked only a few days in the plant when he suddenly develops acute eczema affecting the face, neck, scrotum and less commonly the flexures. The eyes become puffy and there is often an eruption that may weep or crust. The hands and arms are generally unaffected. The condition appears quite suddenly during a working shift or a few hours later and is produced especially by fumes, although fine powder particles may also be responsible. It responds quickly to the removal of the patient from the plant and daily treatment in the ambulance room.

In the second type, the soreness starts between the fingers, on the backs of the hands, the fronts of the wrists and on the forearms. Other parts that may especially be affected are those exposed to friction from clothing, *e.g.* the wrists, ankles, neck and joints. In the third type of dermatitis, the blistering may combine features of the other two types.

The problem of permanent alternative work for dermatitis cases presents difficulties, especially since most cases of dermatitis from formaldehyde occurring in this group of factories become sensitised to it, so that they seem more liable to blister subsequently, but a few develop an immunity or possibly take greater care on returning to their work. Urea formaldehyde resins seem to be as potent a cause of dermatitis as phenol formaldehyde preparations.

The acute effects of formaldehyde vapour on the eyes and respiratory passages are well known; they are frequently produced by vapour concentrations as low as 20 to 30 p.p.m. Lower concentrations may result in watering of the eyes, irritation of the throat and upper respiratory tract. In spite of the vast number of investigations which have been undertaken there is a notable lack of information and agreement on the possible chronic effects in man produced by the repeated absorption of small quantities of formaldehyde over a long period of time.

Polythene

In pure form this has been shown by experience to be remarkably innocuous, having been used for many medical and surgical procedures involving close and prolonged contact with human and animal tissues.

Vinyl chloride

For ease and safety in transport and handling, vinyl chloride is contained in metal cylinders under pressure as a liquid. It is a mild narcotic and concentrations in the atmosphere should not exceed 500 p.p.m. if working conditions are to be considered satisfactory. It boils at −13.9°C. and the liquid will act as a vesicant if it contacts the skin.

Polyvinyl chloride

No cases of dermatitis have been observed in the manufacture, processing or handling of PVC. A few cases, however, have been reported as the result of contact with the final plastic article, which usually contains plasticisers, stabilisers and other substances besides the polymer. The added chemicals in this instance are not primary skin irritants and there have been no skin diseases in the group of factories studied which could be directly attributed to them. Common examples are tricresyl phosphate, phthalates, chlorinated paraffin wax, lead carbonate, cadmium and calcium stearate, pigments, etc.

Plasticisers such as tricresyl phosphate and phthalates that are added to PVC increase its flexibility and it should be appreciated that under certain circumstances these substances may 'bloom' or 'leach' out of the final plastic material. This may be a slow process, during which time the plastic deteriorates and gradually becomes brittle. The toxicity of ortho-tricresyl phosphate is well established, 3 to 5 g. being a fatal dose to domestic fowls. It can be absorbed through the skin.

Other chemicals may also be carried out in this 'leaching' process. Lead carbonate, for example, is sometimes used as a stabiliser for PVC, and lead salts have been found experimentally in sufficient quantities to be detected by quantitative analysis in liquids left in contact with plastic tubing or vessels made of this material.

No cases of poisoning from these added substances have occurred in the factories mentioned.

There are fortunately less harmful chemicals that can be substituted for toxic plasticisers to produce a flexible material. The phthalates are a typical example, since dimethyl and dibutyl phthalate have been used on the human skin as insect repellants. Some of the higher phthalates have been submitted to physiological tests with satisfactory results when they are administered by mouth, by inhalation or applied to the skin.

Methyl methacrylate

This is a colourless liquid (b.p. 100°C.) with a sweet, characteristic odour. It is a mild irritant of the mucous membranes and in a few instances has been known to produce dermatitis. The atmospheric concentration should be maintained below 1,000 p.p.m. if working conditions are to be regarded as satisfactory. Its odour can be detected at far lower concentrations.

Polymethyl methacrylate

The final polymer is comparatively inert, like many others, *e.g.* it is used in the manufacture of contact lenses, artificial teeth, splints and surgical instruments. Its effects at times, however, suggest that the polymer may liberate traces of free monomer or in some circumstances decompose on contact with the skin to form the monomer. In a sufficiently fine state, for instance, the powdered polymer can produce a dermatitis like that due to methyl methacrylate. This has occurred in sieving the polymer in powder form and also as the result of cutting sheets of it with a band-saw, when a spray of fine powder or 'swarf' may be ejected over the hands.

Isolated cases have occurred of workers who complained of coughs, sore throats and 'chestiness' as the result of inhaling fine polymethacrylate powder or fume. Such complaints can easily be prevented by improving the working conditions and particularly by providing local exhaust ventilation.

Methyl α-chloracrylate

When the methyl radical in the α position in methyl methacrylate is replaced by chlorine the result is a compound with markedly irritant properties.

This monomer can be polymerised to

form materials similar to those given by polymethyl methacrylate. Methyl α-chloracrylate (b.p. 126°C.) is a colourless liquid with an odour resembling methyl methacrylate. It is a powerful vesicant whose vapour will produce inflammation of the lung if inhaled in sufficient quantities. A drop or two on the unprotected skin will produce a blister in a day or two, even though the affected part is thoroughly washed within a few seconds of the time of application. Specially designed plants and the most stringest precautions failed to prevent some accidents to those working with this material, although no serious complications developed in any incident. Methyl chloracrylate penetrates rubber, so that protective clothing of this material is inadequate. Some workers were burned in this way from accidental splashes of which they remained unaware because of their protective clothing until the earliest symptoms of smarting and tingling appeared a few hours later or blisters had occurred. An antidote was available for skin contamination in the form of a solution of potassium permanganate (half a dram to the pint).

The vapour of methyl chloracrylate is markedly irritant even in low concentrations (5 to 10 p.p.m.) so that symptoms of eye irritation with inflammation and weeping occur at an early stage. These symptoms may disappear without treatment and, if the worker remains in contaminated atmospheres, he is very likely to develop a severe conjunctivitis, often after a latent interval of a few hours. This can be prevented in almost every case if the eyes are irrigated as soon as the first symptoms manifest themselves.

Polymethyl chloracrylate

The presence of free monomer or decomposition products under particular conditions is suggested by the fact that machining the polymer on a lathe has given off the typical odour of the monomer and in one or two cases has produced eye symptoms. Local exhaust ventilation is sufficient to remove this hazard.

Laminated plastics

The actual impregnation of the filler with the resin may be undertaken by very primitive methods, sometimes only the bare hands being used. Since the resin is usually mixed with the monomer before application, it is not very surprising that cases of dermatitis occur as a result. In some cases, dermatitis may be caused by the fabric itself, as, for example, when woven-glass-fibre cloth may be used for the purpose. It can be prevented by mechanising the process or, less satisfactorily, by providing the operators with suitable protective clothing.

Control valves. Features of the new Mason-Neilan 10,000 series control valves are illustrated and described in a new 8-p. pamphlet issued by Crosby Valve & Engineering Co. Ltd.

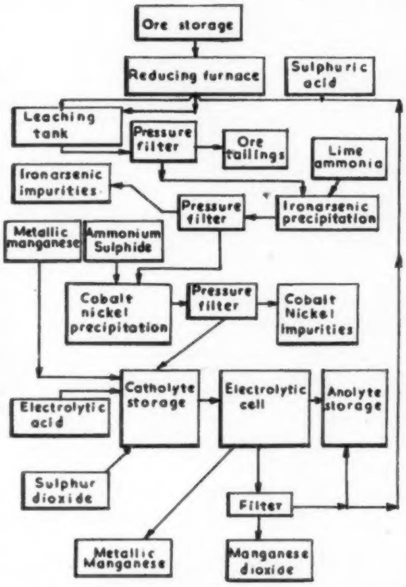
Manganese Production by Electrolysis

MANGANESE occurs chiefly as the dioxide (pyrolusite); other forms, such as the carbonate, silicate, lower oxides and sulphides, are found in smaller quantities. The commonest way of obtaining the metal is by reduction of the ore by heating with carbon. The product obtained contains, among other things, carbon as an impurity. Reduction with aluminium has also been employed to produce pure metal but the cost is high.

The use of the electrolytic process for obtaining manganese from its ores is likely to prove economical and to give a product free from impurities. Electrolytic manganese has been successfully used in the production of non-ferrous manganese alloys, and also in steel production. It is particularly suitable for the production of special steels whose characteristics are greatly affected by the presence of impurities, even in traces.

Commercial production

In the *Journal of Scientific and Industrial Research* (India), 1953, **12A** (10), 457-462, T. Banerjee gives an account of the methods of producing electrolytic manganese which have been adopted in some of the leading metallurgical works in the U.S.A., Canada and India. Among these is the method used at the works of the Electromanganese Corporation, Knoxville, Tennessee, where the electrolytic production of manganese was taken up in 1939. The procedure is as follows:—



Flowsheet for the production of pure manganese (after Hammerquist).

Since manganese ore (with the exception of rhodochrosite) is not soluble in sulphuric acid, it is ground into a fine powder and reduced in an electric furnace in a counterflow of producer gas. The reduced ore is leached with spent electrolyte containing 150 g. $(NH_4)_2SO_4$ and about 25 g. H_2SO_4 per litre and fortified with more ammonium sulphate and sulphuric acid as necessary. The solution is purified by blowing air when ferric hydroxide precipitates out and carries down any arsenic present in solution. If the arsenic content in the electrolyte is high, more iron is added. The liquid after filtration is treated with ammonium sulphide in a lead-lined tank when sulphides of cobalt, nickel, iron and other heavy metals come down. The filtrate contains manganous sulphate, ammonium sulphate, traces of ammonium sulphide and soluble metal sulphides. Sulphur dioxide is now passed through the solution until the concentration of sulphur dioxide reaches 0.1 g./l. Deposition from this solution can be continued for more than 10 hr.

The purified electrolyte is electrolysed in cells made of laminated wood and lined with phenol plastic. Each cell measures $96 \times 28 \times 43$ in. and contains 27 anode and 26 cathode compartments. The latter are made of oak and covered with canvas or cloth diaphragm. The anodes are of lead and the cathodes of stainless steel. The current density (c.d.) is kept at 35 to 40 amp./sq. ft. under 5 v. Electrolysis is continued for 30 to 40 hr. when 12 to 15 lb. of manganese is deposited. The deposit is washed, dried and stripped.

The method has undergone several improvements. The optimum cathode c.d. is 18 to 20 amp./sq. ft. The anolyte contains: manganese (as $MnSO_4$), 3-6; H_2SO_4, 25-30; and $(NH_4)_2SO_4$, 200 g./l. The catholyte contains: manganese (as $MnSO_4$), 24-26; and $(NH_4)_2SO_4$, 200 g./l. The current supplied to the cell is so adjusted that the potential drop is about 1.47 v. at 30°C.

From his survey of this and other methods the author puts forward the following as the criteria for the production of electrolytic manganese.

Choice of electrolyte

Both chloride and sulphate baths can be used. As manganese ores are not easily dissolved by acids, the use of the more active hydrochloric acid may be advantageous. The advantages of chloride baths over sulphate baths are lower power consumption, longer duration of deposition and absence of manganese dioxide deposit.

Against this, chloride baths, being more corrosive, necessitate the use of special construction materials for cells and other containers. Also, objectionable gases, *e.g.* chlorine, are given off and the ammonia concentration in the bath has to be maintained by frequent additions.

The use of the sulphate bath is preferable when the deposition is to be continued for long periods. The optimum concentration of manganese sulphate, in sulphate baths, is about 25 g./l. Ammonium sulphate is a useful adjunct in the catholyte as it gives a bright deposit of manganese and prevents the formation of oxides and hydroxides. Stripping of the deposit is facilitated by the presence of arsenic and copper in the bath.

Controlling the *p*H of the electrolyte, oxidation of metallic impurities by blowing air and addition of sulphides generally suffice to free the electrolyte from undesirable substances.

Electrolytic cell

Canvas-covered wooden frames for encasing the anodes and the use of suspended cathodes are useful developments. Maintenance of a continuous flow of electrolyte from the cathode chamber to the anode chamber is also an advantage; the spent electrolyte coming out of the anode chamber is highly acidic and can be used for the treatment of fresh ore.

To prevent the anodic oxidation of manganese, a suitable diaphragm is necessary. As has been described, wooden frames covered with cloth or canvas can be used with sulphate baths. As canvas diaphragms are attacked by chloride baths, resistant glass-fibre diaphragms coated with inert adhesives or No. 1 Vinyon provide suitable materials.

Electrodes

Lead anodes are satisfactory for use with sulphate baths. The formation of manganese dioxide at the anode is reduced by the use of a lead-silver alloy; addition of arsenic to the bath also helps to reduce dioxide formation. Lead alloy anodes containing tin (9 to 50%), cobalt (up to 4.4%) and with or without antimony (1 to 1.5%) are also satisfactory.

Cathodes of stainless steel have proved satisfactory. The presence of a thin film of silicate on the surface facilitates stripping. Aluminium cathodes covered with aluminium powder have also proved useful.

The *p*H of the catholyte has to be maintained, as nearly as possible, at the neutral point (between 6 and 8). The manganese deposits in this range are free from nodules.

Power requirements

The voltage employed for electrolysis is 4 to 5. Satisfactory deposits are obtained when the current density is maintained at 20 amp./sq. ft.; the current efficiency is about 55% and the power consumption per lb. of manganese is 3.6 kwh.

The current density and time of deposition can be varied over a wide range with only a slight variation in current efficiency, but there is a considerable variation in the power requirement.

The optimum temperature of the cell during electrolysis is about 35°C. Lower temperatures decrease the current efficiency and higher temperatures favour the formation of nodular deposits.

Absorption towers

There were many illustrations of absorption towers and tower fillings at the Chemical Equipment Exhibition at Olympia. This very fine exhibition increased interest in the important processes of absorption and desorption of gases carried out in many chemical plants.

The book* in hand deals primarily with the packed tower, which is most frequently used for such processes. The authors have aimed at providing the engineer with the information about methods, and the data which he needs in order to design full-scale towers which are satisfactory from both the technical and the economic points of view. Design, and the fundamental theories of diffusion and analogies between fluid friction have been stressed, and so the comparison between heat transfer and mass transfer has been omitted. On the other hand, greater emphasis has been laid on the design of liquid distribution systems and on economic factors than would be in an ordinary text-book.

The methods described here are largely based on results of work carried out by Imperial Chemical Industries, Ltd., as is suggested by the publishers' acknowledgment, and much of the work has not been previously published. As the methods have been widely used within I.C.I., it can be assumed that they are an improvement on earlier ideas. The book, therefore, should be most valuable for the practical engineer.

Two design methods are put forward, one making use of absorption coefficients, and the other of transfer units. Various problems are considered, such as that of absorption with chemical reaction, and a collection of design calculations is included, illustrating the application of the methods described, and should prove very useful.

One regret is that in this remarkable book, the question of the engineering materials to be used as packings is not discussed at much greater length.

FELIX SINGER

**Absorption Towers*, by G. A. Morris and J. Jackson. Butterworths Scientific Publications, in association with Imperial Chemical Industries Ltd., 1953, pp. 159, including index. 30s. net.

Phthalic anhydride from tar oils

It is known that phthalic anhydride is obtained by vapour-phase oxidation of alkylnaphthalenes and other polycyclic hydrocarbons. Consequently, the production of phthalic anhydride from coal-tar naphthalene oils was investigated. When a vanadium pentoxide/potassium sulphate/silica catalyst was used no marked deterioration of catalyst was observed in an experiment with naphthalene oil over a period of 28 hr. Operating variables of liquid- and air-space velocity were studied and naphthalene and naphthalene oils of coke-oven and vertical-retort origin compared. The phthalic anhydride yield can be predicted and is dependent on the naphthalene content of the oil and the operating variables.

These experiments, which are described in the *Journal of Applied Chemistry* by J. Shelmerdine, F. Popper and D. McNeil, of the Coal Tar Research Association, showed that the yield of phthalic anhydride from a naphthalene oil is greater than can be obtained from the naphthalene in the oil, even if it could be extracted without loss. At the same time the heat evolved in reaction is considerably increased and, as a consequence, heat removal would present the most important problem in the design of an oxidation plant.

Lime hydration. Hydrated lime should be finely divided, pure and 'sound.' There are two types of unsoundness, one due to particles of unhydrated lime, which cause 'popping' and 'pitting,' and the other to finely divided unhydrated or incompletely hydrated lime, causing 'general expansion.' Soundness is now generally recognised as essential in hydrated lime for the building trade, but there are two other important properties which are seldom considered by manufacturers. They are the chemical reactivity and the plasticity of the hydrate. Both these properties depend upon the nature of the particles formed in hydration and they can be controlled by the process of hydration. It is well known that lime putty made in the usual way by slaking lime in excess of water is more plastic and reactive than hydrate produced as a dry powder, and research has shown that to make the most plastic and reactive hydrated lime it is necessary to approach as nearly as possible to the conditions which obtain in slaking to a putty. This is accomplished when the design and operation of the hydrator is such that the maximum possible portion of the hydration reaction takes place in the presence of liquid water. These points are explained in a 24-page booklet, *Lime Hydrating*, by the Sturtevant Engineering Co. Ltd. The booklet gives a full description of both normal and pressure hydration by the Knibbs-Sturtevant process, with photographs of the various plant units.

MOLECULAR DISTILLATION

The process of molecular distillation does not seem to have become established as a generally recognised method of investigation and treatment of liquids in the low vapour pressure range. A reason for this neglect may be that the technique belongs more to the domain of the physicist, whereas its advantages would probably be better appreciated by the chemist.

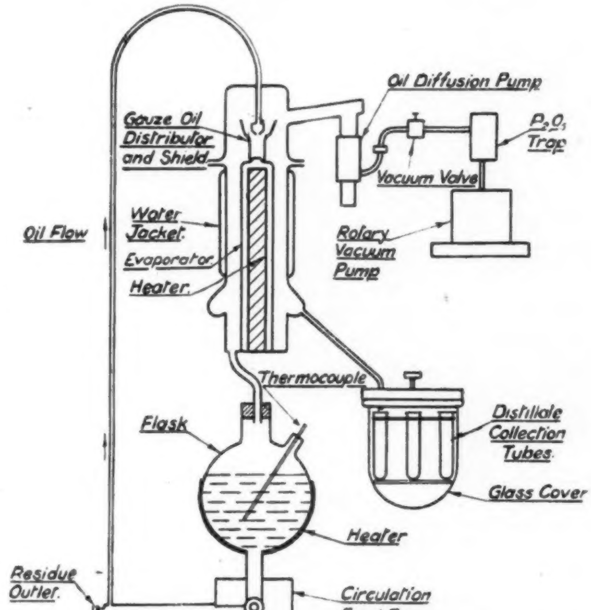
WITH these remarks, G. Burrows, A.M.I.ChemE., opened his paper, 'Some Aspects of Molecular Distillation,' at a meeting of the North-Western Branch of the Institution of Chemical Engineers at Leeds University on December 12.

The influence of the mean free path on the molecular distillation process was discussed, and a theory which attempts to describe the process was explained. The application of the theory to data already published, and also to some recent experimental work, gave values in fair agreement with the experimental results.

Equations which express the degree of separation obtainable in a two-component system were developed, and some test figures were compared with those derived from theory.

The author then described typical examples of batch and continuous molecular stills, together with the precautions to be taken under operating conditions.

In dealing with the operation of the process, he pointed out that, as molecular distillation must be operated in a vacuum of the order of 10^{-3} to 10^{-4} mm. of mercury, it is essential that all joints and glands should be vacuum-tight. The joints may be exposed to comparatively high temperatures and the pipelines may contain oil, so that, for most purposes, the use of natural rubber is excluded. On the small laboratory stills the use of synthetic rubber 'O' rings in machined grooves in the flanges has been found satisfactory, the flanges being water-cooled where necessary. On the larger stills, water-cooled flanges become too complicated and would wastefully remove useful heat, so the flanges and unions are allowed to run hot and oil-sealed metal gaskets are used. Units such as feed and extraction pumps are conveniently sealed by immersing them in a liquid bath, the ordinary gland packing being removed and replaced by three or four silicone 'O' rings gently squeezed together by means of the gland nut. If proper precautions are taken, the maintenance of vacuum causes little or no trouble. On laboratory stills, glow discharge tubes can be used to indicate the degree of vacuum; the one situated between the still and the diffusion pump should remain 'black' during distillation, the other, between diffusion pump and the rotary backing pump, gives, by its appearance, an indication of the kind and quantity of gases or vapours which are being pumped. Where a continuous reading of the degree of vacuum is required, a Pirani gauge unit is situated on the backing side of the diffusion pumps and connected to an indicating instrument.



Circulating batch still.

When operating continuous stills it is important that the feed rate to the evaporator should be maintained constant. If the feed rate increases, the thickness of the liquid film on the evaporator increases, and the rate of distillation is reduced with corresponding variations in the proportions of the fractions being collected. It is equally important that the heat input to each stage should be maintained at a constant value, since, at the usual operating temperatures, an increase in temperature of between 8 and 10°C. doubles the vapour pressure. If the highest degree of separation is required, it is necessary to have a comparatively thin liquid layer, which would also limit the throughput of a continuous still. If the layer is too thick, there may be an undesirably high temperature gradient through it, and also a tendency for the outer portions to become deprived of the more volatile constituents, which will cause a reduction in the degree of separation of the components.

Another aspect of the process which must not be ignored is the thermal efficiency, which is limited by the close proximity of the cold condenser and the hot evaporator. A typical heat balance analysis showed that, of the total heat supplied to a vertical tube still, 60% was lost by radiation, 10% was lost by conduction through the metal parts, 15% was employed in heating the oil to distillation temperature, and 15% was utilised in providing the latent heat of evaporation. These figures are, of course, only an indication of the heat distribution; individual cases will depend on the throughput and material undergoing treatment, and on the design of the still.

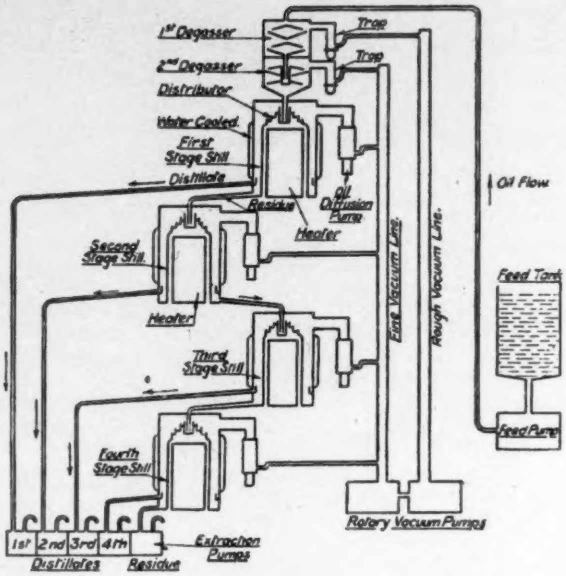
Tray still

This still is usually operated at constant distilling speed, the temperature being progressively increased as the lighter fractions are distilled off. The tray must be kept shallow to avoid too great a depth of liquid, with its attendant troubles of temperature gradients and variations in composition through depletion of the upper layers; the quantity of material which can be satisfactorily treated is therefore limited; the still described has a capacity of about 30 c.c.

Circulating batch still

The initial charge of this unit, about 350 c.c., is contained in a glass flask situated below the evaporator. The liquid is heated and degassed under vacuum while in the flask. When degassing is complete, a small gear pump, situated below the flask, pumps the liquid up through a vertical controllably-

heated pipe and delivers it on to a distributor at the top of the evaporator. The distillate is condensed on the surrounding glass water-cooled condenser, and is collected, as required, in four receivers, each having a capacity of about 70 c.c. The evaporator consists of a thick-walled nickel-plated copper tube heated internally by radiation from an electrical resistance element, the temperature being determined by a thermocouple attached to the wall of the evaporator. The still, degassing flask, gear pump and receiver are all maintained continuously under vacuum by means of an oil diffusion pump. In operation, the liquid is circulated continuously over the evaporator at a constant rate, the evaporator being held at a series of selected temperatures chosen to suit the boiling range of the material. When the distilling rate at a given temperature has fallen sufficiently, the temperature is raised to the next chosen value and simultaneously the next distillate collecting tube is brought below the outlet spout. This procedure, if carried out at low temperatures, improves separation of the components, but may take a long time. A quicker method is to operate at constant distilling speed, with some sacrifice of separation, the distillate collecting tubes being changed at arbitrary intervals.



Four-stage continuous still.

Degasser and devolatiliser

Low-boiling volatiles in the liquid to be distilled can give rise to troublesome splashing and entrainment, leading to a direct transfer of distilland from the evaporator to the condenser. Before attempting a batch distillation with such a liquid it is sometimes advantageous to submit it to a separate preliminary degassing and devolatilising treatment. A devolatiliser of robust construction and capable of dealing with relatively large quantities of liquid has been described in British Patent No. 695,560.

Four-stage continuous still

In a still which has been constructed for continuous operation a first- and second-stage degassing unit is provided before the feed liquid enters the first-stage still. The first-stage degasser consists of an evacuated vessel maintained at a pressure of about 1 mm. in which the incoming liquid can bubble and froth and so release a large proportion of its dissolved gas. Suitable baffles must be fitted so that only the released gas is allowed to enter the pipeline used for evacuation. After passing through a liquid seal the second-stage degasser is reached, where the pressure is about 0.05 mm., and which is designed to break up the liquid as much as possible, and so assist the release of gas, by means of cone-shaped trays which cause the liquid to flow alternately from the centre to the outside and back again, the liquid falling in drops or thin streams from the lower edges of the trays. From the second degasser the liquid flows down through each of the four stages, the residue from any one stage forming the feed for the next stage. Liquid seals are interposed between the various stages so that the pressure in each stage can vary, within limits, without affecting either the preceding or following stages. A temperature indicating instrument on the control cubicle is connected to thermocouples attached to the upper and lower portions of the evaporator of each stage.

The distillates from each of the four stages, together with the residue from the last stage, are extracted continuously by means of separate gear pumps each having a non-return valve on the outlet side. The vacuum is indicated by a Pirani gauge unit situated in the pipeline between the diffusion pumps and the rotary pumps, and connected to an instrument on the control cubicle; as an additional check, a discharge tube vacuum indicator is attached to each still stage.

It is highly desirable that the feed rate and still temperatures should remain constant under operating conditions. The feed pump is therefore of the variable stroke type, and the feed pump motor runs at constant speed, this motor and the still heaters all being fed from a voltage regulator which maintains the voltage to these units constant within $\pm$ 0.5%.

Fractionating still

In an experimental still of this type values of the relative volatility α exceeding the theoretical value for a single distillation have been obtained with the usual E.H.P.-E.H.S. mixture, but further experimental work is needed in order to assess its performance. The achievement of high values of α under conditions of total reflux gives useful information, but the quantity of material obtainable is an important feature from the practical viewpoint. Further work is therefore needed in order to determine the quantitative separation which can be effected under conditions of partial reflux.

Centrifugal still

The inherent advantages of a thin film combined with a short heating time are of special benefit when treating heat-sensitive materials. Few facts are available to show whether these features lead to higher rates and better separation than can be obtained with stationary stills. Before a comparative evaluation can be made, more information of a quantitative nature is required. Combined fractionating and centrifugal stills have been described (B.P. 644,621, U.S.P. 2,606,146).

Applications of molecular distillation

In this paper it has been assumed that re-evaporation from the condenser is negligible. If the condenser temperature is 20°C., this implies that the vapour pressure of the condensed film at that temperature should not be much greater than 10^{-5} mm., a condition which sets a limit to the scope of the molecular distillation process. As a rough guide, liquids having molecular weights of 300 or more condense satisfactorily at 20°C. Butyl phthalate (molecular weight 278) is an example of a liquid which, with care, can be molecularly distilled, but if too high a distilling rate is attempted the apparatus becomes filled with a 'cloud' of vapour. In general, as the molecular weight increases, the vapour pressure decreases and the rate of distillation falls correspondingly. With organic substances, decomposition usually begins in the range 300 to 350°C., so that this sets an upper limit to the temperatures at which distillation can occur. If the vapour pressure in this upper temperature range is so low that the rate of distillation is negligible, the liquid cannot be distilled. Liquids having

a molecular weight of about 1,000 approach this condition, although materials having mean molecular weights somewhat higher than this value may sometimes be satisfactorily distilled.

No similarity is exhibited by liquids when they are molecularly distilled; degassing may occur easily and quietly with little bubbling, or it may be difficult, the bubbles bursting almost explosively, with splashing. When a component of comparatively high volatility is present in a liquid of much lower vapour pressure, the lighter component tends to vaporise readily as the temperature of the mixture is raised and to form small bubbles or pockets of vapour which rapidly expand and burst, causing contamination of the distillate; this trouble can sometimes be remedied by a preliminary devolatilising treatment.

Substances which are usually readily amenable to treatment by molecular distillation include high-boiling petroleum derivatives, such as lubricating oils and petroleum jellies. Animal and vegetable oils, which may undergo polymerisation when heated, frequently give rise to a layer of gum or gel on the evaporator surface after a time. The extent and rate of formation of the layer can often be reduced by careful operation, but some materials in this class seem to be particularly liable to the trouble, which occurs even when a centrifugal still is used. The removal of the layer from the evaporator by mechanical means appears to be a possible remedy. Mechanical removal of ice from the surface of the condenser during high vacuum sublimation has been described by R. S. Morse (*Chem. & Ind.*, Oct. 9, 1948, S. 13).

Recent publications

Swing-hammer shredders, for reducing fibrous materials to coarse or reasonably fine products, are described in a new 14-p. illustrated brochure by British Jeffrey-Diamond Ltd. Materials which can be treated include angelica root, bark, copra, cork, corn cob, fruit peel, ginger and liquorice root, paper, peat, town refuse, straw, tobacco, wood refuse and others. The machines can also be used as primary units where multi-stage reduction is necessary to obtain really fine products. The operation and construction of the machines are described and specifications given, with the dimensions of the various models.

Design and construction in steel plate. Whessoe's publication No. 53 describes the company's activities in the oil, gas, chemical, steel and other industries. The introduction, which takes the form of a brief survey of the Whessoe organisation with a pictorial representation of the shops' layout at Darlington, stresses the fact that steel plate and its handling to provide capital plant for the industries mentioned has been the theme behind the post-war reorganisation which has taken place. In the brochure are illustrated and described the various forms of capital plant manufactured, ranging from storage tanks of every size, including those for vapour conservation applications, to tank fittings manufactured under licence from the Shand & Jurs Co. It covers heat exchangers, refinery plant, pressure vessels and various forms of processes and storage methods devised for the gas, coke oven, steel and allied industries.

Carbon and graphite, with their wide variety of thermal, chemical and physical properties, have long been of interest to the chemical and process engineer as a material for the construction of plant handling corrosive fluids. Various applications of *Carbinert* impervious carbon and graphite are described in a new 6-p. illustrated folder by the Morgan Crucible Co. Ltd. A graph shows the heat-transfer characteristics for a tube bundle made in this material, and there is a list of some of the reagents which have been tested against *Carbinert* and found satisfactory. A table shows the approximate physical properties of the material for chemical engineering applications.

Copper-manganese-tin alloys. The metallography and mechanical properties of copper-base alloys containing 1.5 to 23% manganese and 4 to 14% tin have been investigated and form the subject of an illustrated booklet which has been issued by the Tin Research Institute. It is found that manganese reduces the primary solid solubility of tin in copper, particularly below the eutectoid temperature. The tempering of quenched alloys below this temperature results in the precipitation of a new phase, designated δ'.

The effect of heat treatment on the quenched alloys, with or without previous cold working, has been examined, and it is found that good mechanical properties are obtained if the quenched alloys are cold worked before tempering. In the absence of cold work, considerable brittleness may develop on tempering, owing to the preferential precipitation of δ' at the grain boundaries or along crystallographic planes.

The mechanical properties appear to be governed more by the total manganese-plus-tin content than by the contents of the individual metals, although, of the two alloy additions, tin seems to exert more influence than manganese. Within the composition range investigated, it is possible by suitable treatment of the alloys to obtain ultimate tensile strengths of up to 30 tons/sq.in. with elongations of up to approximately 45%. The tensile strength can be further increased to a maximum of about 40 tons/sq.in., though above 33 tons/sq.in. there is a progressive and fairly rapid fall in the elongation.

Alloys containing not less than 15% manganese with not less than 6% tin are almost white in colour.

Export Opportunities

Sugar mills

The erection of four new sugar mills at Erzurum, Erzincan, Elazig and Malatya, in the eastern provinces of Turkey, is under consideration and tenders may be called for early next year. Interested firms should approach Seker Fabrikalari T.A.S., Genel Müdürlügü, Atatürk Bulvari, Ankara.

Water supply installations

Tender Notice No. 2, issued by the Directorate General of Municipalities, Ministry of Interior, Government of Iraq, calls for tenders for the supply, erection, construction and complete installation of flocculation and settlement tanks, rapid precipitant mixers, chemical sterilising apparatus, rapid pressure filters, chemical dosing apparatus, pump and power house building, overhead travelling crane, electrically operated centrifugal pumping plant, suction and rising mains, elevated service tank, modifications and renovation of existing elevated service tank, station sewage disposal system and all ancillary works included in the new water supply scheme for Kut to provide approximately 1 million gal./day.

Tender Notice No. 3 is for the supply, erection, construction and complete installation of the settlement tank, alumina dosing apparatus, precipitant flash mixer, rapid pressure filter, air blower, gaseous chlorinator, electrically operated centrifugal pumping plant, suction and rising mains, elevated service tank, pump house building and all ancillary works comprised in the water supply scheme for Yousufiya of 65,000 gal. capacity per day.

Tenders should be submitted by registered post in sealed envelopes endorsed 'Tender for Kut (or Yousufiya) Water Supply Scheme' and addressed to the Directorate General of Municipalities, Ministry of Interior, Baghdad, Iraq, to arrive before noon on April 15 in the case of the Kut project and April 29 in the case of Yousufiya.

The general conditions and specification for either of these two projects can be obtained from the Accountant of the Directorate of Municipalities, or the Royal Iraqi Embassy in London, on production of a valid Chamber of Commerce Certificate, price I.D. 5/000 or £5 per copy.

Acetone and isopropyl alcohol

Karl O. Helm, Hamburg 36, Esplanade 38, wish to act as agents for a U.K. manufacturer of acetone and isopropyl alcohol.

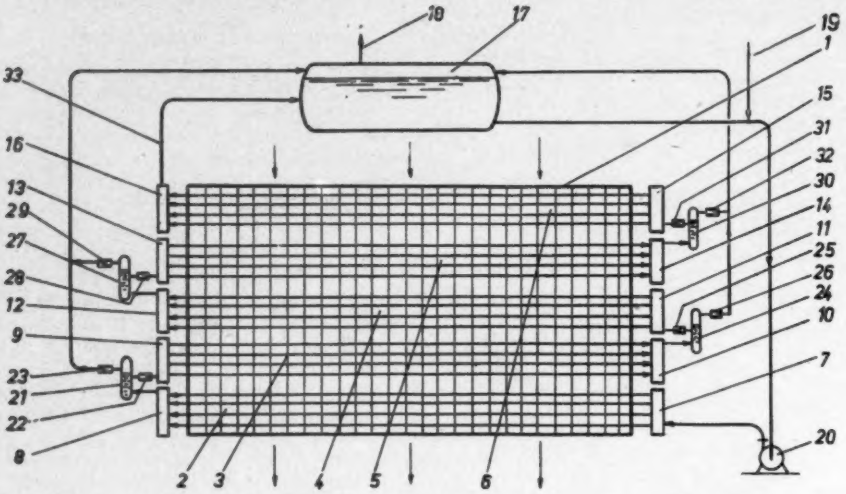
U.K. manufacturers who take action on this enquiry should notify the Board of Trade (see below).

For further details of these enquiries, write to the Export Services Branch, Board of Trade, Lacon House, Theobalds Road, London, W.C.1.

Recent German Patents

Fractionated cooling of contact ovens

In the apparatus described, synthesis gases are supplied at the top of the oven 1 and the synthetic products are drawn off at the bottom. The cooling medium flows through the panel assemblies of cooling pipes 2 to 6. The individual pipes are inserted at their ends into the chambers 7 to 16. Above the oven there is a vapour pressure, when the pump 20 conveys into the pipe chamber 7 with exceptionally high starting pressure. The commencing pressure of the cooling means prevails in the pipe assembly 2. The throttle valve 22 eases it to a reduced pressure with which the pipe system is operated. The throttle valves 25, 28, 31, effect further reduction of pressure up to a final pressure which



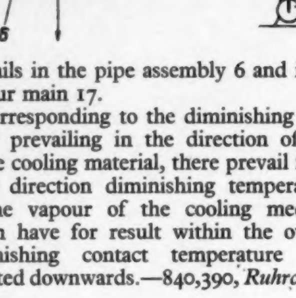
main 17 to which the vapour produced (especially steam) is taken through a pipe 18. Fresh cooling material is fed through the pipe 19 corresponding to the vapour loss. A pump 29 sucks the cooling medium through the vapour main 17 to force it into the pipe chamber 7, whence it flows through the pipe assembly 2 into the pipe chamber 8 opposite and thence into a vapour separator 21, of which the liquid space is in communication with the vapour chamber 9 through a valve 22 and the vapour space thereof through a valve 23 is in communication with the vapour main 17. From the assembly of pipes 3 the cooling medium proceeds to the pipe chamber 10 and thence to the vapour separator 24, which allows the liquid cooling medium to flow on through a valve 25 into the vapour chamber 11, whilst the vaporised part of the cooling medium flows directly to the vapour main 17 through a throttle valve 26. Coming from the pipe chamber 8 the cooling medium thereafter flows in sequence through pipe assembly 4, pipe chamber 22, vapour separator 27, throttle valve 28, pipe chamber 13, pipe assembly 5, pipe chamber 14, vapour separator 30, throttle valve 31, pipe chamber 15, pipe assembly 6, and finally to pipe chamber 16. It is conveyed hence through a pipe 33 back to the vapour main 17. The throttle valves 29 and 32 conduct the vapour existing in the cooling systems 4, 5, into the collecting receptacle 17.

Through the throttle valves 22, 25, 28, 31, the cooling material can be retained in the pipe system 2, 3, 4, 5, 6, at diminishing prevails in the pipe assembly 6 and in the vapour main 17.

Corresponding to the diminishing pressures prevailing in the direction of flow of the cooling material, there prevail in the same direction diminishing temperatures of the vapour of the cooling medium, which have for result within the oven a diminishing contact temperature fractionated downwards.—840,390, *Ruhrchemie A.G.*

Frothless conveyance of frothing chemical reactions

The wall 3 dividing the input chamber 1 from the reaction chamber 2 has an opening 4 at its bottom, which connects the two chambers. A rotary pump 5 is disposed in the opening 4. The chamber 2 has an overflow outlet 7 of which the height of the overflow is adjusted by means of a slide 8. The materials fed to the chamber 1 go through the opening 4 into the lower part

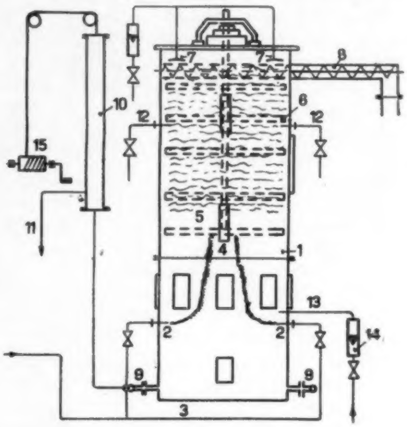


of the chamber 2 where the reaction takes place.

The apparatus can function with or without overflow from the chamber 2 over the partition 3. In the first case the height of the overflow 7 is so arranged by the slide 8 that the surface of the liquid in the chamber 2 during operation remains below the upper edge of the partition 3.—838,444, *S.A. des Manufactures des Glaces et Produits Chimiques de Saint-Gobain, Chauny & Cirey.*

Countercurrent precipitation of organic colloids

The cylindrical container 1, having a diameter of 1 to 3 m. and a height of 6 to 12 m., is provided at its lower part with six to eight nozzles for the introduction of the solution made frothy with gas, which is fed to the nozzles through the annular pipe 3. The froth leaving the nozzles coagulates in the space which is filled with means for producing precipitation. The coagulated material, as a result of its gaseous content, rises proportionately quickly through the space 4 to the boundary layer 5 where the foam coagulates. Through a longitudinally-extending pipe



arrangement 6 the coagulated layer is continuously conveyed away, to avoid the formation of canals in the coagulated material. Fresh precipitation material is added through the nozzles 7 at the top of the apparatus and runs downward through the coagulated layer. The coagulated material can be conveyed away through the worm conveyor 8 for pressing and drying at the bottom of the precipitation apparatus. The excess of precipitation means flows away continuously through the pipes 9, 11, and through overflow pipe 10 actuated by rope tension and the windings 15. Through nozzles 14 at the top of the apparatus additional material, *e.g.* acid, may be supplied. Excess chemicals are washed out of the coagulate in the upper part of the apparatus by precipitation. It is possible to adjust the desired *p*H value for the precipitation with acid or lye, which is delivered at 13 through the measuring apparatus 14.—839,194, *Bachmann.*

Plant and Equipment

Semi-paste mixer

A new mixer is claimed to be capable of performing the heaviest work, thoroughly mixing pastes, paints, enamels, toothpaste, and other viscous or semi-liquid substances. The mixing blades revolve in one direction, the container in another, so that the entire contents of the vessel come under the action of the blades. When it is required to free the container, little effort is required to raise or lower the blades, as they are balanced by a counterweight in the main column.

The mixer is of the 'change container' type. Standard containers are reinforced at the brim and provided with trunnions or with lifting handles for easy transportation about the shop. Tubs of wood or other material, according to the nature of the substances to be mixed, can, however, be supplied by the makers, Steele & Cowlishaw Ltd. All containers are interchangeable and extra units are available.

If it is desired to discharge the mixture while the blades are revolving, the container may be brought to rest by means of a clutch which disengages the driving wheels. As the machine is self-contained, it can be arranged for direct coupling to an electric motor, or be fitted with fast and loose pulleys for belt driving. All gear wheels are machine-cut from the solid, and completely enclosed. Ball and thrust bearings to the base supporting the container ensure quiet running and low power consumption.

The mixer, known as the semi-paste mixer, Type G, is made in a range of sizes from 10 to 60 gal. working capacity.

Quick-response voltage recorder

A new voltage recorder, made by Evershed & Vignoles Ltd., is of the continuous roll chart type and contains two movements which are essentially d.c. recording voltmeters having a rapid response and negligible pen-to-paper friction. The maximum torque produced by the pen movement is approximately 7 oz. ins.

The recorder is servo operated and works in conjunction with an amplifier unit, the amplifier and recorder being connected by a 5-ft. long 18-core flexible cable with plug and socket ends. The input to the recorder is opposed by the feedback voltage from a resetting potentiometer which is controlled by the position of the pen. The error, being the difference between these voltages, is amplified and drives the pen movement until the feedback voltage balances the input. The pen zero may be set in any position and a solenoid operated time marker pen is also included.

The recorder is contained in a portable hard wood case measuring 18 x $11\frac{1}{2}$ x 7 in. and having a canvas carrying strap. The front of the case is mounted on hinges which are so arranged that the whole cover may be removed. The used chart may either be stored on a rewind spool on the clock or ejected through a slot in the front cover. An opening in the back of the recorder case facilitates chart replacement.

The maximum sensitivity may be set to suit requirements between the limits of ± 10 v. to ± 4 v. for a deflection of $\pm 1\frac{7}{16}$ in. It may be continuously reduced down to zero by means of a potentiometer control on the front panel of the amplifier unit. Provision is made to switch this control out of circuit if desired. The input impedance is approximately 50,000 ohms with the sensitivity control potentiometer in circuit and approximately 2 megohms with it switched out. The voltage across the resetting potentiometer is stabilised and the sensitivity is thus independent of mains variations.

It is claimed that the instrument will record sine wave voltages up to 15 c/s with an amplitude error not exceeding 15% for a peak-to-peak amplitude of $1\frac{1}{2}$ in. At smaller amplitudes the frequency response is proportionally higher.

Induction heating system for process vessels

The Wild-Barfield induction heating system has for some time been applied with great success to the heating of metals for forging, billet heating and heat-treatment.

Results of research and development in the heating system over the last few years have made it possible to apply the system efficiently to heating kettles, autoclaves, stills and other process vessels commonly used in the chemical and allied industries. Kestner Evaporator and Engineering Co. Ltd. have acquired the exclusive rights of the system for such applications in the chemical and allied industries.

This patented method of heating employs standard mains frequencies and, it is claimed, eliminates the need for complicated frequency changers which have hitherto been associated with induction heating systems. The simplicity of operation and the close control of temperature attainable are among the features which put this method of heating ahead for many of the processes carried out in the fields concerned.

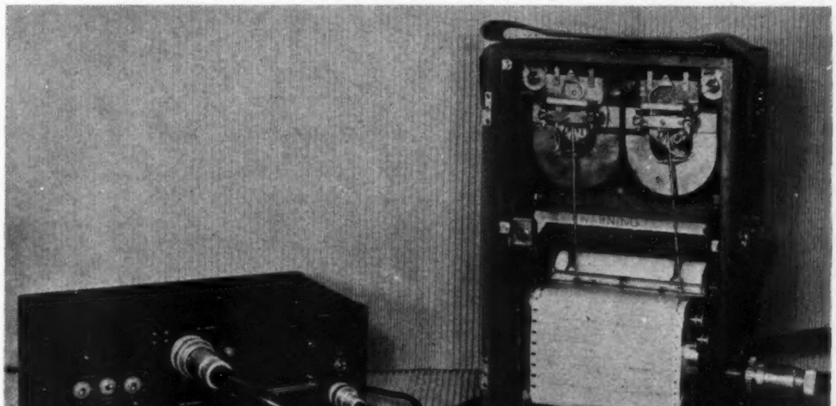
With Wild-Barfield's research facilities and wide knowledge on the electrical side combined with the chemical engineering experience and pilot plant laboratories of the Kestner company, the best advice on the application of this new method of heating is assured. Kestner will shortly have available an installation in their laboratories where tests and demonstrations can be made.

Pilot-operated pressure controller

The design efficiency of many industrial plants is often only attainable by close control of a number of variable factors, of which the supply pressure of steam for heating, for example, or of fuel to burner equipment, is of considerable importance from the viewpoint both of economy and plant performance.

Most reducing valves of the simpler type do not maintain an exact controlled pressure unless the flow is steady, and for precise control the more costly pilot-operated regulator is often the only solution. The Fisher Governor Co. Ltd. are now offering an inexpensive pilot-operated regulator—the *Wizard*—which, they claim, provides accurate control of vacuum or pressure of all fluids and gases up to 5,000 p.s.i.

It has been specifically designed for use with the company's range of diaphragm



New voltage recorder of the continuous roll chart type.

motor valves, using the same operating medium—either compressed air or clean gas at a pressure of 25 p.s.i. or above. The measuring element consists of a stainless steel Bourdon tube for pressures above 15 p.s.i., or, for lower pressures, of a bronze or stainless steel bellows. A simple flapper and nozzle arrangement controls the air pressure sent out to the diaphragm motor valve and the measured pressure change required to promote full valve movement—proportional band—is readily adjustable between 2% and 50%, according to the plant characteristics.

A convenient set point adjustment is provided, but being of the non-indicating type it is cheaper for many applications than conventional indicating and recording pressure controllers.

Improved centrifugal pumps

The Sigmund *PumPak*, the latest addition to the extensive range of centrifugal pumps manufactured by this firm, introduces a new standard in the design and performance of close coupled electric pumping sets. All the proved best features of integral construction are incorporated in this new unit with additional refinements developed by the company.

The two principle features which distinguish the *PumPak* from all other close coupled pumping sets are the Sigmund stator pack service, and the special ventilation arrangement.

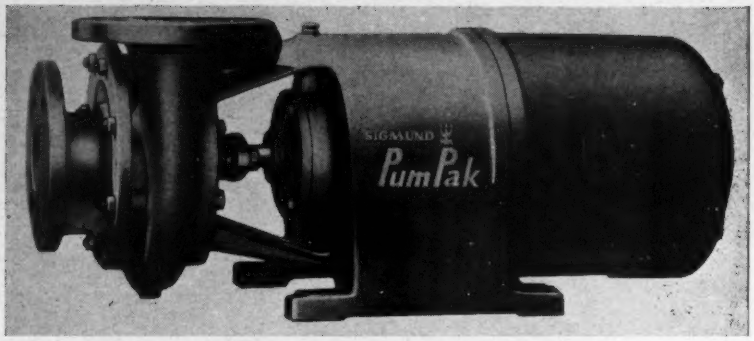
In the usual type of integral pump the difficulty of replacing the stator in case of electrical breakdown is often the principal objection to the application of a close coupled unit. The patented method of withdrawing the stator, and the special stator pack replacement service, completely obviate this objection. The stator housing is so designed that it can be removed complete with stator without disturbing the pump in any way, it being only necessary to disconnect the leads to the terminal box and remove four nuts to enable the whole unit to be completely withdrawn.

Stocks of each size of *PumPak* stators are held at the works so that in the event of an electrical failure developing in the set, a replacement stator can be despatched promptly thereby enabling the pump to resume duty with the minimum of delay, inconvenience and expense.

The withdrawal and replacement of the *PumPak* stator can be carried out in a matter of minutes as the form of construction obviates any possibility of misalignment.

The importance of adequate ventilation has been specially studied, and the cooling arrangement is another patented feature. Cool air is drawn through the entire motor, by a fan mounted on the pump side of the stator, a stream of which circulates around the pump side bearing, effectively cooling the bearing, and preventing the entry of any liquid into the bearing housing.

The initial *PumPak* range now in full-scale production covers units from $1\frac{1}{2}$ to 15 h.p. at 1,450 and 2,900 r.p.m. with capacities up to 500 g.p.m. and heads up to 175 ft. giving a wide selection of units, but an additional series of fan-cooled and small types will shortly be available.



The drip-proof version of the new Sigmund PumPak range of electric integral pumps.

Electronic sulphur indicator

An instrument which can detect sulphur in concentrations as low as 0.1 p.p.m. was described in a paper presented to the American Chemical Society recently by H. Landsberg and E. E. Escher, of Consolidated Engineering Corporation. The *Titrilog*, as it is called, is a chemical-electronic instrument which, it is claimed, can record within 20 to 30 sec. any changes in sulphur concentrations.

The operation of the instrument is based on a titration. In the case of sulphur, the titration is done with bromine. This is electrolytically generated in a solution in which the sulphur compounds are absorbed from the gas. A feed-back amplifying system controls the bromine generating current so that the rate of bromine generation is at all times equivalent to the rate of absorption of the sulphur compounds. A meter then records the generating current, which is always proportional to the sulphur concentration in the incoming gas stream being tested.

Among the industrial applications of the *Titrilog* described were the following:

In **natural gas**—to test the hydrogen sulphide concentration at removal plants, and to control the concentration of sulphur compounds when they are used in natural gas, which is odourless, as a warning agent for gas leakage.

In **personnel protection.** The instrument may be used to monitor plant atmospheres where sulphur compounds may reach toxic levels. It is equipped with an alarm for this use.

As a **catalyst protector.** Small tracers of sulphur compounds often reduce the efficiency of catalysts. The *Titrilog* can be used to determine the source of the sulphur and corrective steps can be taken.

In **air pollution** studies. A helicopter-borne *Titrilog* was used recently to determine concentrations in the vicinity of industrial chimneys.

In **odour research.** In a chlorophyll test, garlic and onion oils, both of which are sulphur compounds, were studied. The effectiveness of chlorophyll in cutting down the odour was accurately measured by the instrument, it is claimed.

The *Titrilog* is a rectangular unit $5\frac{1}{2}$ ft. high, $22\frac{1}{2}$ in. wide and $18\frac{1}{2}$ in. deep. The chart recorder is mounted at eye level. Below it is the control panel. There is also a portable model weighing only about 50 lb.

Fine filter

The *Stellar* filter has been designed for very fine filtrations. It is used in the production of fine chemicals, insulin, liver and similar animal extracts and other injection solutions, all of which must be completely free from any suspended matter.

The filter consists essentially of a cylindrical shell and outlet manifold constructed to take wire-wound elements or candles, which are fine strainers and are employed as the foundation for a deposited filter bed of specially graded kieselguhr or kieselguhr mixed with other materials.

The element consists of a tube with equally-spaced longitudinal ribs having a screw thread cut along the entire length, providing on each rib a series of grooves in which the wire, wound in consecutive turns on the ribs, is firmly located. The openings over the whole area of the element are of regular size, ensuring an even coating of filter aid.

For general purposes with aqueous liquids, filter shells are made of mild steel and lined when necessary with a hard plastic enamel. For foodstuffs and corrosive substances stainless steel is used. The filter elements for water, oils and non-corrosive liquids are made with extruded-brass tubular cores, electro-tinned and wound with either Monel or stainless-steel wire. Where the residual liquid must be conserved, special arrangements can be offered according to the nature of the application, *e.g.* syphon-type elements, a reversible filter on trunnions or a separate small filter for dealing with the contents of the main filter. Paterson Engineering Co. Ltd. are the makers.

For further particulars of plant and equipment please use the coupon on page 102.

World News

GREAT BRITAIN

Power-Gas to supply world's largest blast furnace

Eight new blast furnaces on order from the Power-Gas Corporation—including one for the Steel Company of Wales which will be the largest in the world—should augment the country's yearly pig-iron production by 2.2 million tons. This was stated by the chairman and managing director of Power-Gas, Mr. N. E. Rambush, at the company's annual general meeting. The orders booked by the group during the year amount to £6¼ million, which is slightly less than the peak figure of last year. The value of orders received from the iron and steel industry is the highest in the company's history.

Two further specialities have been added to the company's range of products through a preliminary collaboration with two overseas firms—namely Chemiebau-Zieren for sulphuric acid plants and General American Transportation Corporation for Wiggins floating roofs for petroleum oil storage tanks.

The company have resumed a pre-war arrangement for the manufacture and sale in Japan of their specialities under licence.

Power-Gas are increasing their investment in their Australian subsidiary by extending their present subscribed capital from £A10,000 to £A75,000 to assist them with their larger turnover. A closer co-operation with the Perry Engineering Co., Adelaide (in which the subsidiary company holds a block of shares) has been arranged.

Conference on steam

It is expected that important new projects will be announced at the forthcoming annual engineers' conference of Spirax-Sarco Ltd., to be held at the Palace Hotel, Buxton, from April 12 to 15. The conference will include technical papers and discussions on subjects such as hot-air drying installations; condensate drainage and air venting of steam-heated drying cylinders; heating and process steam considerations at breweries, gas works, hospitals, etc.; and thermostatic control problems in steam and hot-water installations and certain low-temperature plant. The conference will be attended by representatives and associates from many overseas countries.

I.Chem.E. symposium on gas absorption

A big programme has been arranged for a symposium on gas absorption to be held from April 5 to 7, inclusive, by the Institution of Chemical Engineers at the University, Birmingham. The facilities of the University will be available for meals and, in addition, a limited amount of accommodation in student hostels has been secured. The registration fee, including a set of preprints, is £1 1s. for members and £2 2s. for non-members. There will be four sessions; the following are the dates and times, with the names of the chairmen:

Monday, April 5, 2.30 p.m., Prof. F. H. Garner (Chairman, Chemical Engineering Group, S.C.I.).

Tuesday, April 6, 10 a.m., Mr. Stanley Robson (President of the Institution); 2.30 p.m., Mr. E. J. Dunstan.

Wednesday, April 7, 10 a.m., Dr. B. Edgington (Chairman, Midlands Branch of the Institution).

Change of name

The Birmingham Welding Co. Ltd., engineers, fabricators and special process plant builders, have decided to adopt their registered name of 'Birwelco' as the company title and are now to be known as Birwelco Ltd. The change was decided upon as a matter of convenience and does not imply any change in the organisation of the company.

A new development which coincides with the change of name is the formation of a subsidiary company, Brown Fintube (Great Britain) Ltd., for the manufacture under licence of the American Brown Fintube heat exchangers and heaters.

Dispersed pigments

A year ago Acheson Colloids Ltd., makers of colloidal dispersions of graphite, carbon and other materials, launched a Dispersed Pigments Division to deal with increasing requests from industry for high-grade dispersions of pigments and other finely divided solids. In 1953 the Division, which has its factory at Slough, Bucks, experienced unusual success in what is something of a pioneer idea in Britain. It has been customary for makers of leathercloth and unsupported PVC sheeting, and undertakings who need colours, to disperse their own pigments in plasticisers and other media. With 50 years' experience in dispersing and stabilising solids it was logical that Acheson's should put that knowledge at the disposal of the plastics, paint and associated industries.

In consequence their plant at Slough, designed to disperse pigments in all media, has been working to capacity for a large number of users of dispersed colours. New ideas have been adopted and, using careful laboratory control, high standards have been achieved.

During 1953 undertakings in various parts of industry called on Acheson's to prepare stabilised dispersions of carbon blacks, titanium dioxide, mineral and organic colours of every kind, in media ranging from straight plasticisers through treated oils and resins to simpler carriers such as alcohols, ketones, hydrocarbons and water. Among those who have farmed out their pigment dispersing this way are prominent makers of leathercloth, lacquers, leather paints, PVA flooring, coloured millboard, plastic cables and acetate sheeting.

Standard reference block for ultrasonic testing

The F.E. 18 Ultrasonics Panel of the British Welding Research Association is considering the need for a standard, single-hole, steel reference block for use in ultrasonic testing. Such blocks would need to be commercially available and should bear a mark showing that they are of approved design and quality.

All firms and individuals interested in this possibility are asked to communicate with the Secretary, the F.E.18 Committee, British Welding Research Association, Abington Hall, Abington, Cambridge, from whom further information may be obtained.

Intensive chemical engineering course

A four-week intensive practice school in chemical engineering is to be set up by the Ministry of Supply at the Royal Ordnance Factory, Bridgwater, Somerset, during the summer of 1954. The school will be open to honours students in their final university year. The course will be limited to 12 students, who must be British subjects and who will be selected by competitive interview. During their stay in Bridgwater they will study problems relating to chemical plant, under the guidance of Mr. E. S. Sellers, M.A., M.Sc., A.M.I.Chem.E., of the Department of Chemical Engineering, University of Cambridge. They will also meet technical staff from the factory for group discussions.

No fee will be charged for the course and students will receive an allowance of £4 2s. 6d. per week while the school lasts, with free travel to and from their homes. Hostel accommodation near the factory will be available at a cost of about £2 15s. per week.

Provisional dates for the course are from August 16 to September 13, 1954. If the school is successful this year, it may be continued as an annual event.

Application forms are obtainable from the Director of Ordnance Factories (Administration and Finance), Room 88, Ministry of Supply, Leysdown Road, Mottingham, London, S.E.9. They must be returned before March 31.

Insignia Award in Technology

The City and Guilds of London Institute have reported on the progress that has been made since their Insignia Award in Technology was established some 12 months ago. The object of the award is 'to provide a high qualification for persons in industry whose initial training was based primarily upon practical experience combined with theoretical study and who, having gained appropriate City and Guilds' certificates as craftsmen or technicians, have now advanced in their industry by a combination of progressive experience and further study.' Candidates, who must be at

least 30 years of age, are required to write a thesis, and, if this proves satisfactory, to be interviewed by a special panel.

In connection with the inauguration of the scheme, and to set the standard of achievement to which it is hoped future C.G.I.A.s will aspire, the Council have, in each of the five industrial groups, made up to three Foundation awards to distinguished representatives of industry who, in their early days, had gained City and Guilds certificates. These were bestowed at the last yearly meeting of the Institute.

A copy of the regulations governing the award, together with notes for the guidance of candidates, can be obtained from the Director, Department of Technology (I.A.), City and Guilds of London Institute, 31 Brechin Place, London, S.W.7.

EUROPE

O.E.E.C. to study petrochemicals and dyestuffs industries

Permanent working parties to exchange information in the field of petroleum chemicals and dyestuffs are to be established by the Chemical Products Committee of the O.E.E.C. It is also proposed to set up an *ad hoc* working party on statistics. These decisions were made at a recent meeting of the committee at which the programme of work for 1954 was discussed.

The Committee, in common with other O.E.E.C. technical committees, meets as a rule six times a year for a session of three or four days. The intervals between meetings provide an opportunity for the Secretariat and such working parties as the Committee may appoint to prepare material for the plenary sessions of the Committee.

The work of an O.E.E.C. technical committee falls into two broad divisions. Statistical studies are prepared of the current position and future prospects of those sections of European industry covered by the committee concerned; and the committees are available to undertake special investigations as required by the O.E.E.C.

BELGIUM

Cement factory begins production

A new cement factory with an output capacity of 40,000 tons/year has begun production on the shores of Lake Tanganyika, near Albertville, in the Belgian Congo. Built by the *Cimental* company, the new plant will manufacture cement by the wet process.

Output of cement in the Belgian Congo last year amounted to little more than 200,000 tons.

YUGOSLAVIA

Chemical expansion plans

Yugoslavia's production of chemicals for civilian use is to be stepped up 20% this year. Several new factories are planned, including one to produce 60,000 tons p.a. of artificial fibre.

★ PERSONAL PARAGRAPHS ★

★ **Mr. W. L. James** has been elected chairman of Mellor Bromley & Co. Ltd., Leicester, following the recent death of Mr. H. R. Backhouse (chairman and managing director). He joined the company in 1920 and was made a director in 1933. On the formation of Pegson Ltd., Coalville, in 1932, as a separate member of the group to build mining, quarrying and road-making plant, he became a director of that company and, later, chairman and managing director. In the early days of Pegson, Mr. James was responsible for the introduction into this country of specialised American machines to be manufactured under licence.

★ **Mr. G. A. Hannah** has been appointed managing director of Pegson Ltd. in succession to Mr. James. He joined Mellor Bromley in 1938, but within a few months transferred to Pegson Ltd. as general works manager. In 1945 he was promoted to director.

Within a few hours of the announcement of his appointment Mr. Hannah left Coalville on a business trip to Singapore, Kuala Lumpur, Sydney, Melbourne, Wellington, Auckland, San Francisco and New York.

★ **Mr. G. L. R. Pearce** has joined Enamelled Metal Products Corporation (1933) Ltd. as technical representative on the chemical side, succeeding **Mr. A. R. Wyatt,** who has now left the company.

★ **Mr. T. H. Hilton,** of Joseph Crosfield & Sons Ltd., has left the U.K. on a four months' tour of the Caribbean area with the object of expanding the company's export sales of sodium silicate, the bottle-washing detergent *Solgon* and allied products.

★ **Mr. C. W. Engelhard** has relinquished his duties as president of the Engelhard Industries Group. He continues as chairman and managing director.

★ **Mr. G. V. Richdale** has been elected president in place of Mr. Engelhard. He comes to his new position after 14 years in the gold-mining industry of South Africa, where he was a director of the Rand Mines Group. He is of British birth. The companies of which he becomes president include American Platinum Works, Amersil Co. Inc., Baker & Co. Inc., Charles Engelhard Inc., East Newark Realty Corporation, Hanovia Chemical & Manufacturing Co., Irvington Smelting & Refining Works and Nieder Fused Quartz Co., all of which are located at Newark, U.S.A.; also the D. E. Makepeace Co., Division of Union Plate & Wire Co., of Attleboro, Mass., and the National Electric Instrument Co. Inc., of Elmhurst, Long Island. This group includes Baker Platinum Ltd., in London, one of the principal refiners and workers of precious metals, and manufacturing and sales subsidiaries in Paris, Copenhagen, Zurich, Milan, Johannesburg, Melbourne, Tokyo, Bogota, Rio de Janeiro and Toronto.

★ **Air Commodore Sir Frank Whittle,** F.R.S., the British pioneer in the field of jet propulsion, has taken up an appointment with the Bataafsche Petroleum Maatschappij, The Hague, one of the principal operating companies of the Royal Dutch/Shell Group. He will give advice in the field of mechanical engineering relative to the development of techniques and equipment in the petroleum and chemicals-from-petroleum industries. He has already familiarised himself with the activities, including the work of the research laboratories, of the Royal Dutch/Shell Group.

★ **Mr. C. G. Graham Hayman,** chairman since April 1953 of the management committee of The Distillers Co. Ltd., which controls all the activities of the D.C.L. organisation, was made a Knight Bachelor in the New Year's Honours List. For the past 30 years Mr. Hayman has been very closely connected with the distilling and industrial interests of D.C.L. He became a director of the parent company in 1936. Before assuming his present responsibilities he was chairman of the D.C.L. Industrial Group executive committee, which has carried through the company's extensive programme of post-war industrial expansion. He represents D.C.L. on the boards of National Chemical Products Ltd. (South Africa) and of C.S.R. Chemicals Pty. Ltd. (Australia), whose new chemicals plant at Rhodes, N.S.W., we described in January. He is, in addition, chairman of the British Tyre & Rubber Co. Ltd. From October 1950 to October 1953 he was chairman of the Association of British Chemical Manufacturers.

NETHERLANDS

New flax process

A new chemical process claimed to replace the normal retting process in flax treatment and to save about 40% in time and money has been developed by two research workers of the Netherlands Institute for Applied Natural Science Research.

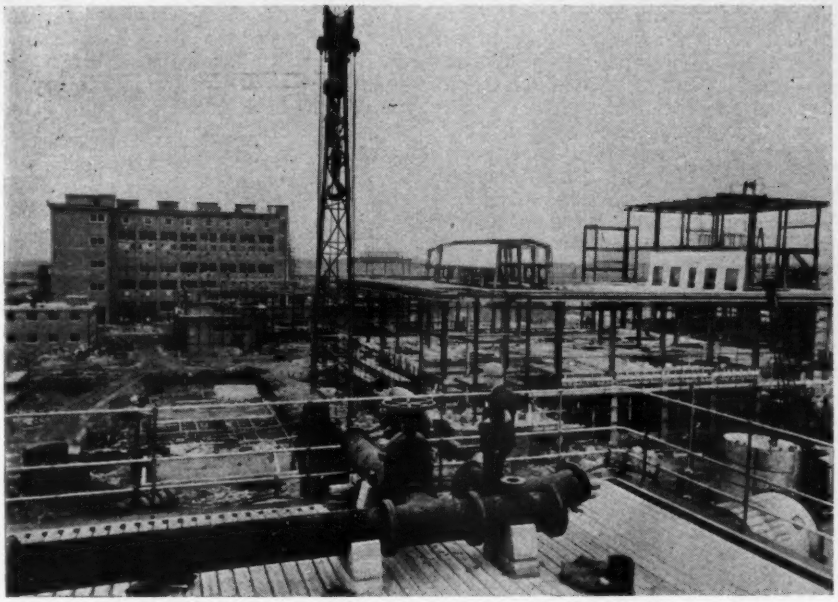
At the present moment the Netherlands' annual export of flax, straw, yarns and fibres totals about 90 million guilders, but application of the new method is expected to result in an increase in exports to about 150 million guilders.

A factory in which the new process will be put into practice is shortly to be established in Delft at a cost of about 1½ million guilders.

HUNGARY

More fertilisers to be imported

Big demands for fertiliser imports may be expected from Hungary over the next three years under a farm development plan costing £362 million just ordered by the



NIGHT AND DAY WORK ON 'TERYLENE' FACTORY

This photograph shows construction work on the polymer section of I.C.I.'s new 'Terylene' fibre factory at Wilton, North Yorkshire. As report in our August 1953 issue (p. 261), I.C.I. are doubling the size of the original project. Total investment is nearly £20 million. Construction of the first section of the factory started in July 1952; work is going on night and day and is up to schedule. Three million bricks and thousands of tons of structural steel and concrete are going into the construction of the plant. Specially designed processing machinery will be installed to produce the 'Terylene' polymer and the final products—filament yarn and staple fibre. The first unit, with a capacity of 11 million lb. of 'Terylene' p.a., will be completed by the end of this year and production will begin early in 1955. In 1956 the second unit of the plant, which will double this output, will come into operation.

Government. The Ministers of heavy industry, home and foreign trade are made responsible for seeing that farms get 348,000 tons of fertiliser in 1954, 490,000 in 1955 and 600,000 tons in 1956. Of this, 45% must be nitrogenous.

The Ministry of Light Industry is directed to produce 30,000 tons of superphosphate and mixed fertiliser in 1954, rising to 100,000 tons in 1955. Granulated superphosphate must be put on the free market at 15s./cwt.

FINLAND

Cellulose exports

Finland has already sold 230,000 tons of 1954 production of cellulose. This means that the whole export production of the first quarter has been disposed of. Sales to Britain, the biggest customer, have been made at firm prices.

Last year, Finland exported a total of 790,550 tons of cellulose, compared with 715,550 tons (revised) in 1952. The total for 1953 comprised 468,128 tons of sulphite cellulose and 322,422 tons of sulphate cellulose. Britain took 270,763 tons of Finnish cellulose in 1953, compared with 288,763 tons in 1952. Exports to the United States amounted to 106,476 tons and to France 104,139 tons. Western Germany bought 51,790 tons.

Production of cellulose in 1953 amounted to 1,132,108 tons, compared with 1,156,108 tons in 1952. The Finnish cellulose industry will have a production capacity this year of 1,500,000 tons, but despite the favourable demands this capacity will not be fully employed.

NORWAY

Experimental atomic power plant

The Norwegian-Netherlands research group at the Atomic Institute at Kjeller, Norway, now have plans for building an experimental atomic power plant. It is proposed to build the plant in Holland at an estimated cost of £1¼ million. The research and preparatory work necessary before construction can start is to be carried out at Kjeller, where the Norwegian-Netherlands team are jointly operating a uranium reactor which is now producing radioactive isotopes on an increasing scale (see CHEMICAL & PROCESS ENGINEERING, 1953, **34**, (10), 333-334).

FRANCE

First styrene monomer plant

The Koppers Co., of the U.S., is to furnish engineering services and field assistance in the building and operation of a new styrene monomer works in France, the first of its kind in that country. It will be erected at Mazingarbe for the Societe Houilleres-Pechiney-Progil, will take two years to build and will have a capacity of 14,000 metric tons p.a.

SOUTH AFRICA

Production of cheaper fertiliser begins

The first significant shipment of nitrogen fertiliser made in the Union recently left the Somerset West factory of Cape Explosives Ltd., which is part of the African Explosives & Chemical Industries Group. A factory official said that the Somerset West plant had been working all out and this full production was being achieved in the face of certain initial difficulties.

The factory, together with another factory at Umbogintwini, in Natal, is receiving considerable quantities of ammonia from the big new ammonia plant of African Explosives at Modderfontein. This ammonia is combined with phosphate in the form of ammoniated superphosphate. With its 3% of nitrogen and 18½% of phosphoric oxide the ammoniated superphosphate closely resembles the Mixture E fertiliser at present favoured by Cape Western Province wheat growers. Mixture E has 1% more nitrogen but 4½% less phosphoric oxide and should therefore cost considerably less than the new fertiliser. So cheap, however, is the ammonia nitrogen made in South Africa from air and water compared with imported nitrogen that the ammoniated superphosphate will actually sell for less than Mixture E.

CANADA

New oil refinery

The Imperial Oil Co. Ltd. has announced plans for the construction of a large oil refinery near Halifax, Nova Scotia. Under its new building programme the company will replace most of its existing plant at Imperoyal, near Halifax, with a new refinery of almost double the present capacity.

The building programme will cost between $25 and $30 million. The new refinery will incorporate the latest developments in processing oil, including the largest fluid catalytic cracking unit in Canada, with a capacity of 27,000 bbl./day.

The daily capacity of the new plant will be 41,625 barrels, compared with a present capacity of 22,000 barrels. Construction of the new refinery will not start until early in 1955, however; completion is scheduled for the summer of 1956.

PHILIPPINES

Electric battery factory

The Philippine Base Metals Co. and one of the country's biggest manganese exporters have announced that a 1-million-pesos flashlight and radio battery plant will be established in Manila in the next few months. Four Japanese technicians will organise the factory.

The first of its kind to be established in the Philippines, this factory will have an eventual output of 10,000 flashlight batteries and 5,000 radio batteries a day. Machinery costing 500,000 pesos is scheduled to arrive from Japan in April.

UNITED STATES

Du Pont's new neoprene plant

E. I. Du Pont de Nemours & Co. have announced preliminary plans to build a new plant to manufacture neoprene synthetic rubber at Montague, Michigan. The plant, tentatively scheduled to begin construction late this year and to be in partial operation by early 1956, is expected to cost about $15 million.

Du Pont's supply of the synthetic now comes from its Louisville, Kentucky, plant, where an expansion project is also under way.

New oil projects

The Great Northern Oil Co. is planning to build the largest oil refinery in Minnesota. The refinery, to be located about midway between St. Paul and Hastings on the Mississippi River, will have a capacity of 25,000 bbl./day and will cost about $20 million. It will process crude oil from the Fosterton Field of Saskatchewan in the Williston Basin.

The Warren Petroleum Corporation is to build a $2-million petrochemical plant near Conroe, Texas. It is designed for an annual output of about 2½ million lb. of pentaerythritol, 4 million lb. of methyl alcohol and 2 million lb. of acetaldehyde.

Petrochemical boom in the South

Petrochemicals, backbone of the Southern United States' growing $3,000 million chemical industry, have given the South an economic uplift unparalleled in history, O. A. Colten of the Shell Chemical Corporation, New York, told the American Chemical Society's Regional Conclave in the Jung Hotel recently.

The South, producing two-thirds of the nation's oil and three-fourths of its natural gas has attracted 85% of the petrochemical industry and as a result is enjoying a 'true renaissance,' said Mr. Colten.

A great movement of chemical industry to the South and South-west has been taking place since the mid-thirties. According to Mr. Colten, more than twenty major chemical companies within the past fifteen years have recognised the advantages of going south: proximity to raw materials (including salt, sulphur, clays and oyster shells), climate suited to economical out-of-doors plant design, ample labour, and favourable access to rail and water transportion.

The nation's great chemical industrial expansion, paced by the South and sparked by petrochemicals, was attributed by Mr. Colten to two factors: new chemical processes based on relatively abundant and inexpensive raw materials and changes in income distribution which have widened the consumer market.

Synthetic fibres are one outlet of the expanding petrochemical industry. Rayon and acetate fibres have been produced from a number of sources, but in recent years, Mr. Colten asserted, material of petrochemical origin has been dominant in the production of rayon, acetate fibres, nylon acrylics, vinyls, and glass protein fibres.

Petrochemicals are also the source of synthetic rubber and many plastics. Thirty years ago, when the petrochemical age was born, plastics output was less than 10 million lb. p.a.; today the U.S. uses 2,500 million lb. p.a. Mr. Colten predicted a rosy future for this industry, with an annual demand for plastics of 9,000 million lb. by 1975.

Synthetic ammonia is one of the major products of the petrochemical industry with a bright outlook. About two-thirds of the ammonia production will go into fertilisers (barring war), either used directly or converted to compounds such as ammonium sulphate or ammonium nitrate.

New factory will make 'Terylene' raw material

The Hercules Powder Co. is to begin immediate construction of a $4-million factory for the manufacture of D.M.T. (dimethyl terephthalate), the basic chemical used in the manufacture of *Terylene* synthetic fibre.

The factory will supply D.M.T. for the $22-million *Terylene* factory now being built by Imperial Chemical Industries of Canada Ltd., at Millhaven, Ontario. It will be built on the site of the present Hercules synthetic resin facilities at Burlington, New Jersey. The Hercules factory will be the first to make D.M.T. by a new air oxidation process. It will have an annual production capacity of about 12 million lb., and will be designed so that this can be increased as other markets for D.M.T. are developed. The factory will supply demands of producers of *Terylene* in foreign countries. It is expected to be in operation in May, 1955.

New polythene plant

The Allied Chemical and Dye Corporation started operations at its new Niagara River petrochemical plant at Tonawanda, New York, which will be devoted to the manufacture of polythene products for waxes and coatings. The plant will utilise gas produced from fuel oil in contrast to the conventional method (in the U.S.A.) of producing polythene from natural gas. It will have capacity to make 20 million lb. p.a. of polythene.

Fertiliser plant in operation

The Davison Chemical Corporation has started production of sulphuric acid at Bartow, Florida. This is the first step in putting into operation the company's $12-million triple superphosphate plant. The rated capacity of the acid unit is 550 tons/day of 100% sulphuric acid.

Japanese pulp firm in Alaska

Representatives of the Alaska Pulp K.K., a Japanese investment company set up last August, have completed the organisation of a lumber and pulp enterprise for the development of forest resources in Alaska.

A spokesman of the company said the new firm, named the Alaska Lumber and Pulp Company Incorporated, had a $1 million capital. It would set up a factory in Sitka to produce lumber and pulp. The Japanese branch would import these products and sell them in Japan.

New petrochemicals plant

The Sun Oil Co. has opened a $15-million petrochemicals plant at its biggest refinery in Marcus Hook, near Philadelphia. The plant has an annual production capacity of 19 million gal. of benzene, the same quantity of toluene and 15 million gal. of mixed xylenes.

Testing methods for plastic pipe

Methods for the evaluation of plastic pipe will be established in an extensive engineering research programme which began recently at Battelle Memorial Institute, Columbus, Ohio. This programme is being sponsored by 29 companies in the Society of the Plastics Industry Inc. These companies are extruders of various types of plastic pipe and suppliers of the raw materials. The programme will develop effective test methods on such factors as bursting strengths, safe working pressures, long-range serviceability of plastic pipe under static pressure and dynamic loading, such as occurs with water hammers and reciprocating pumps.

Owing to the increasing demand for plastic pipe since it first appeared on the market 12 years ago, the number of installations has grown tremendously. Plastic pipe sales for 1953 were expected to exceed $15 million, compared with a $500,000 market in 1948. Various raw materials are at present being used in plastic pipes for transporting water in potable water supply systems; natural gas; oil; mine drainage; chemicals and beverages. It is also being used extensively in varied irrigational installations. While these installations, which, in some cases, constitute field tests, have proved satisfactory, the plastic pipe manufacturers recognise that reliable test methods and engineering data must be developed. Materials from which Battelle will evaluate test methods for plastic pipe are cellulose acetate butyrate, polyethylene, polystyrene and polyvinyl chloride.

The development of these test methods for the evaluation of plastic pipe is but one constructive step in the programme of plastic pipe manufacturers to assure a quality product. At the present time they are also developing standards which it is planned will be issued through the Commodity Standards Division of the United States Department of Commerce, as voluntary industry standards on dimensional weights, sizes and performance specifications for various types of plastic pipe.

A Steering Committee comprising pipe extruders and material suppliers is co-operating with the Battelle laboratories in the development of these test procedures which it is believed will take approximately two years.

A.C.S. president-elect

Prof. Joel H. Hildebrand of the University of California, chemist and educator, has been chosen president-elect of the American Chemical Society. Dr. Hildebrand, who also has won wide recognition as a photographer, mountaineer and one-time manager of the United States Olympic ski team, will head the Society in 1955.

This year's president is Prof. Harry L. Fisher, head of the department of rubber technology in the University of Southern California.

AUSTRALIA

U.S. abrasives company starts production

With an initial investment of around A£250,000, Minnesota Mining and Manufacturing (Australia) Pty. Ltd. is starting operations in Australia. The company now has buildings covering 65,000 sq. ft. on a 24-acre site at St. Mary's, an industrial area. Initial output will include coated abrasives, paper and cloth; cellulose tape, masking tape and other industrial pressure-sensitive tapes; underseal rubberised coating; adhesives; sound recording tape; and reflective sheeting.

The American parent company also has factories in France, Germany, Britain, Canada, Mexico and Brazil.

New oil refinery under construction

Construction work has begun on a new £20 million sterling oil refinery, situated at Kurnell, near Sydney. Australian Oil Refining Ltd., a subsidiary of Californian Texas Oil Corporation (Caltex) is carrying out the work. The latter is jointly owned by the Standard Oil Co. of California and the Texas Oil Co. and has an 80% share in Western Australia Petroleum (Pty.), the company which recently struck oil at Exmouth Gulf in Western Australia.

The new refinery will have a two-stage distillation unit, a propane decarbonisation unit, and a modern fluid catalyst cracking unit.

NEW ZEALAND

Heavy water scheme cancelled

The proposal to develop heavy water from New Zealand's field of geothermal steam in the Wairakei area (see CHEMICAL & PROCESS ENGINEERING, 1953, **34** (11), 339) has been cancelled for the time being. The Prime Minister, Mr. Holland, said that the United Kingdom Government, with whom the New Zealand Government had planned to develop this project, had advised that it did not wish to participate. Therefore no further action will be taken by the New Zealand Government in the matter.

Mr. Holland said New Zealand would concentrate all efforts now on the development of electric power from the geothermal (hot springs) area. He did not disclose the British Government's reasons for withdrawing. 'New Zealand's interest in possible production of heavy water here was primarily because of the contribution that local production might make to Commonwealth defence,' he said. 'It is possible that future geothermal power projects may be of interest to the U.K. Government in connection with the production of heavy water.'

He added that liaison would be maintained with them on the development of geothermal steam resources 'in case there may be some future requirement for heavy water which it will be practicable to supply from New Zealand sources.'

The Leonard Hill Technical Group—March

Manufacturing Chemist — Synthetic Spasmolytics; Phenyl Ethyl Alcohol; Synthesis of Adrenocortical Hormones, 2; Industrial Methods of Sterilisation; Cosmetic Industry; Industrial Applications of Calcium Hydride; Paper Chromatography and Electrophoresis; Progress Reports: Perfumery, Detergents, Antibiotics.

Building Materials Digest—Lightweight Concrete, 2; Fibre Building Boards, 2; Progress in Yugoslavia.

Food Manufacture—The Milling Industry, 2; Vitamins, 1; Processing of Passion Fruit.

Paint Manufacture—Priming Paints for Light Alloys; Paint Technology and the Law; The Physics of Colour; Aniline Inks; 16-mm. Film and the Paint Industry.

Fibres—Concerning Sisal Products; The Making of Brushes; Flax Gives Fillip to an Australian Town; Wool Mothproofing Agents and their Application, 2; Fibres Used in Rope Manufacturing; Venezuela Expands Sisal Growing; Radioactive Isotopes in Wool Research.

World Crops—Chemical Weedkillers; The Cashew Nut; Characterising Spraying Nozzles; American Maize Rust in Africa; Irrigation in Algeria.

Petroleum — Modern Practice in Natural Gas Dehydration; Kuwait; Seismic Prospecting in Deep Oceans; Industrial Petroleum Chromatography, 3.

Atomics—Sources of Neutrons for Experimental Purposes; Nuclear Power Production and Utilisation, 2; Equipment for Production of Neutrons with a 1.5 MeV Accelerator; Non-metallic Materials of Construction for Handling Radioactive Wastes; Radiography for Industry; New Mobile Radiographic Unit.

Muck Shifter—Grand Contour Canal, 2; Geophysical Exploration; Excavator Power Unit.

TURKEY

Engineering contracts for French firms

The French heavy-engineering firm Fives-Lille has been awarded contracts worth about 4,000 million francs in Turkey. The orders are for the building of two sugar refineries for the Turkie Seker Fabrikalari S.A. and for four cement factories for the Turkish Government.

The Société Francaise de Constructions Mécaniques has also been awarded a contract for the construction of a sugar refinery in Turkey, the company discloses.

JAPAN

Japanese glycerin for Spain

Japanese soap manufacturers are negotiating the export of 300 tons of dynamite glycerin to Spain, according to the Japan Soap Manufacturers' Association. Trade circles said these exports were being contemplated in view of growing stocks of glycerin at leading soap plants. They would be made at prices substantially below the domestic prices of glycerin.

The soap manufacturers expect to ship a total of 250 tons of crude glycerin (80% glycerin content) shortly. Under the present plan, 200 tons would be exported to the U.S. and 50 tons to the U.K.

BOLIVIA

New chemical plant

The Chacur Group of U.S. and European businessmen has signed agreements for the installation of an explosives, chemical products and match factory in Bolivia. The Bolivian Government will hold the majority of shares. The Chacur Group has also been negotiating the building of a tin smelter in Bolivia. However, it is believed that the Bolivian Government intends to study closely the technical and economic aspects of such a smelter before resuming negotiations with the group.

MEXICO

Sulphur production developments

The Mexican Sulphur Co. S.A.'s new sulphur plant at San Cristobal, Vera Cruz, has started operations. Sulphur is being produced by the Frasch process and it is the first such operation outside the United States. The expected production rate is 200,000 tons p.a.

Meanwhile, the Texas International Sulphur Co. has entered a contract with Central Minera S.A., of Mexico City, to undertake exploration and development of 123,550 acres of Government concessions held by Central Minera in the State of Vera Cruz.

INDIA

Increase in plastic moulding

There are now 80 firms engaged in plastic moulding in India, as against 40 in 1948. The industry is centred in Bombay and Calcutta. Total private investment is estimated at Rs. 60 million.

About 40 moulding firms are on the active list of the development wing of the Ministry of Commerce and Industry. These firms possess 180 compression presses with a total capacity of 10,200 tons; 263 injection moulding machines with a total capacity of 550 oz.; and 41 extruders of sizes ranging from 1 to 4½ in.

Three engineering concerns in India are making some of the simpler types of moulds. There has been considerable increase in the production of compression and injection moulded goods. In 1948 production was 0.37 million gross; by 1952 it had risen to 1.54 million gross.

Electric lighting and fitting accessories,

buttons, bottle caps, containers for a variety of purposes, and industrial accessories like automobile parts, jute bobbins, jockey pulleys and radio cabinets are some of the articles so far made by the compression and moulding process. Toilet and stationery, household goods such as bangles, toys for children, plates, and industrial articles like umbrella handles and wall tiles have been produced by injection moulding. There has been successful establishment of manufacture during recent years of consumer goods such as toothbrushes, spectacle frames and fountain pens. Production of mechanised toys and metallising plastic articles were also developed during this period.

PAKISTAN

New fertiliser and cement plant

Construction of a fertiliser factory and a cement plant with U.S. and Canadian financial participation was expected to begin at Daudkhel in the Punjab last month. The Governor-General was to lay the foundation stones on February 20, and it is hoped that the cement factory will be in production by the middle of 1955 and the fertiliser plant by early 1956.

The U.S. has contributed $12 million towards the fertiliser factory, which will produce 50,000 tons of ammonium sulphate per annum.

Canada is collaborating with the Pakistan Industrial Development Corporation in the construction of the cement factory, and has contributed $5.5 million under the Colombo plan. The plant will produce 100,000 tons of cement per annum.

VENEZUELA

A proposal is reported to set up a new petrochemical industry, with mixed U.S. and Venezuelan capital, to utilise the natural gas which is at present almost all "flared off." The new company is to have an initial capital of $35 to 40 million, and the plant is to be installed in the Maracaibo Lake area. It will produce fertilisers and other by-products.

MEETINGS

Institution of Chemical Engineers

March 10. 'Design of Pressure Vessels and the Mechanism of Failure in Service,' by A. H. Goodger, 6.30 p.m., the University, Birmingham.

March 13. 'Description and Uses of a Research Experimental Plant,' by H. J. Thurlow, 3 p.m., College of Technology, Manchester.

April 5-7. Symposium on 'Gas Absorption,' the University, Birmingham.

Society of Chemical Industry

March 24. 'Some Recent Advances in Physical Chemistry,' by Prof. A. R. J. P. Ubbelohde, 6.30 p.m., Institute of Metals, Grosvenor Gardens, London, S.W.1. Joint meeting with the R.I.C.

April 5-7. Symposium on 'Ion Exchange and its Applications,' University of London, Gower Street, London, W.C.1.

Chemical Engineering Group

March 18-19. 'Chemical Engineering in the Food Industry,' Wellcome Research Institute, 183 Euston Road, London, N.W.1.

Chemical Society

March 15. 'Chemistry, Biochemistry and Isotopic Tracer Technique,' by Dr. G. J. Popjack, 6.30 p.m., Chemistry Lecture Theatre, the University, Leeds.

March 18. 'Patent Specifications regarded as Chemical Literature,' by Dr. W. A. Sylvester, 7 p.m., Department of Chemistry, the University, Bristol. Joint meeting with the R.I.C. and the S.C.I.

March 24. 'Some Aspects of Oligo- and Polysaccharide Synthesis,' by Dr. E. J. Bourne, 7.45 p.m., Department of Chemistry, University College, Dublin. Joint meeting with the R.I.C. and the S.C.I.

March 25. 'Organometallic Compounds containing Fluorocarbon Radicals,' by Prof. H. J. Emeleus, 7.30 p.m., Chemical Society's Rooms, Burlington House, Piccadilly, London, W.1.

Royal Institute of Chemistry

March 22. 'Chemical Problems associated with Nuclear Reactors,' by J. M. Fletcher, 7 p.m., Dartford Technical College, Lowfield Street, Dartford.

Institute of Fuel

March 19. 'Coal Science and Industry,' by Dr. D. T. A. Townend, Loughborough College.

March 23. 'Solar Energy for Water- and Space-Heating,' by Dr. H. Heywood, 5.30 p.m., Institution of Mechanical Engineers, Storey's Gate, St. James's Park, London, S.W.1.

April 2. 'Total Gasification of Coal,' by Dr. F. L. Dent, 6 p.m., South Wales Institute of Engineers, Park Place, Cardiff.

Institute of Metals

March 8. 'Powder Metallurgy,' by D. D. Howat, 6.30 p.m., Institution of Engineers and Shipbuilders in Scotland, 39 Elmbank Crescent, Glasgow, C.2.

March 11. 'Titanium—A Metal of Engineering Importance,' by Major P. L. Teed, 7 p.m., Liverpool Engineering Society's Rooms, The Temple, Dale Street, Liverpool.

Institute of Petroleum

March 19. Symposium on 'Metal-working Oils,' 2.30 p.m., Institution of Electrical Engineers, Savoy Place, Victoria Embankment, London, W.C.2.

Institute of Welding

March 18. 'Hydrogen *versus* Welding,' by C. L. M. Cottrell, 7.15 p.m., Sun Hotel, Chatham.

Institution of Rubber Industry

March 10. 'Effects of High-energy Radiations on Rubber and Plastics,' by Dr. Charlesby, 7.30 p.m., Polygon Hotel, Southampton.

Manchester Association of Engineers

March 26. 'Engineering Aspects of Glass,' by Dr. R. E. Bastick, 6.45 p.m., Engineers' Club, Albert Square, Manchester 2.

INTERNATIONAL CONFERENCES

March 24-26. Symposium on 'The Chemistry and Physics of Synthetic Fibres,' Society of Chemical Industry, 56 Victoria Street, London, S.W.1.

March 24-*April* 1. The 125th meeting of the American Chemical Society, Kansas City, U.S.A.

March 25-26. Annual meeting of the Swedish Paper and Cellulose Engineers, Stockholm, Sweden.

April 6-9. Conference on 'The Physics of Particle Size Analysis,' Nottingham.

April 8-13. The 38th annual exhibition of scientific instruments and apparatus, Physical Society, 1 Lowther Gardens, Prince Consort Road, London, S.W.7.